METALS AND ALLOYS

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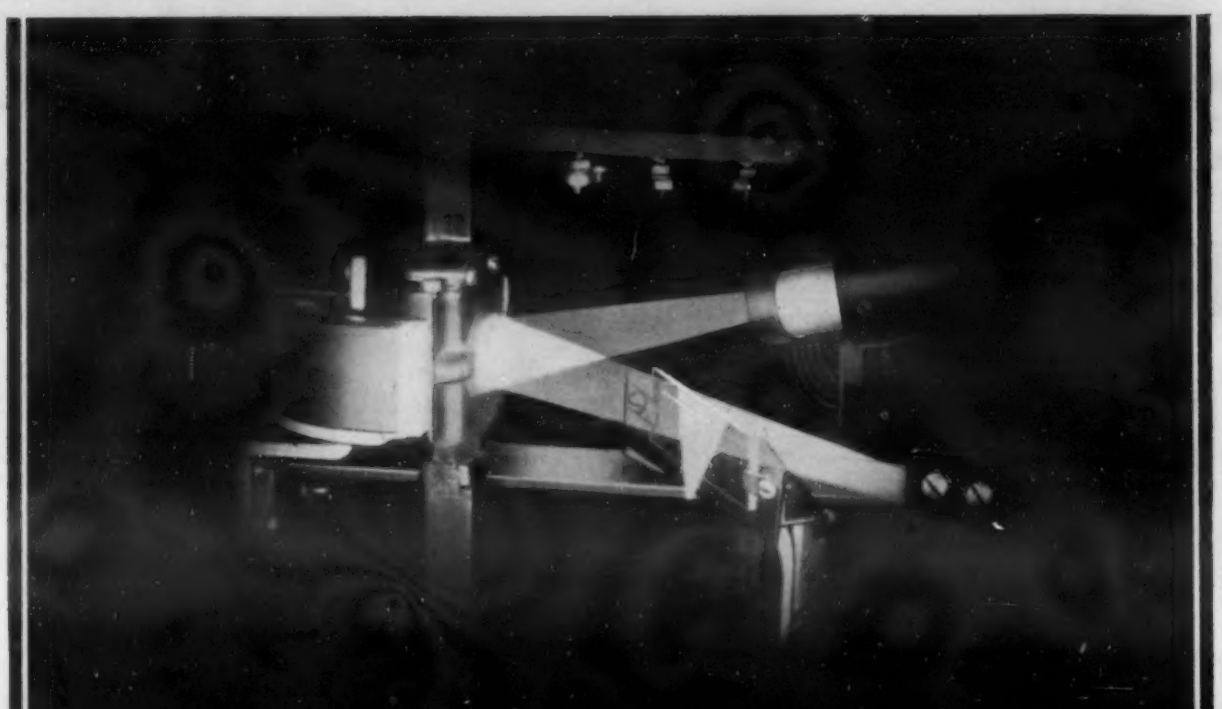

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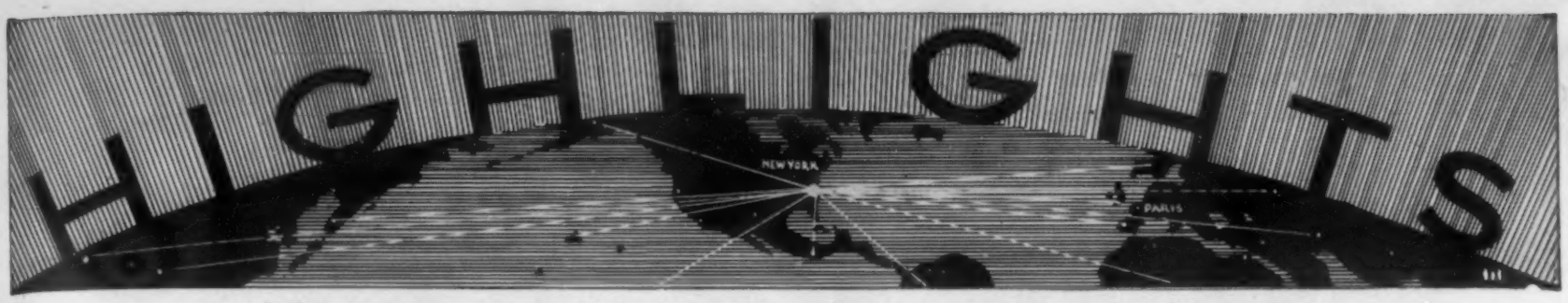

Written by the Abstract Section Editors and the Editorial Staff

DO YOU want to know what metallurgical engineers are saying, the world over? Look in the Current Metallurgical Abstracts. Here are some of the points covered by authors whose articles are abstracted in this issue.

Flaky Steel

The relation between hydrogen and flaky steel is discussed by Drescher and Schäfer (page MA 340 R 5) and by Honda and Hirone (page MA 365 R 2).—H.W.G.

Barnyard Refuse

This, according to Geissel (page MA 340 R 7), is still used in cores in German foundries. We didn't use to call it that when we were on the farm, however.—H.W.G.

Age-Hardening Gets Some More Study and Needs It

Wassermann (page MA 346 R 5) discusses aging phenomena of Al alloys, Tamaru (page MA 346 R 2) reports on the aging of brass in high vacuum and *Metal Industry* (London) reviews the recent researches on the mechanism of age-hardening (page MA 346 R 4.)—O.E.H.

WPA Has Overlooked a Bet

Bräuer (page MA 353 L 4) says the average life of a church bell is 300 years, after which it develops fatigue cracks and must be welded up. Church-bell-welding would be a good occupation for those who like a long rest between their tasks.—H.W.G.

Hydrogen in Steel

German metallurgists are devoting very considerable attention to this subject. It has recently been shown (page MA 356 L 2) that the presence in the steel of certain "hydride formers" such as As, Sb, P, Bi, as well as S and its companions, Se and Te, promote the rate of diffusion of hydrogen through steel during electrolytic pickling. However, according to another German investigation (page MA 357 R 7), such hydrogen is not responsible for the formation of pin hole perforations in the tin coating on tin plate, which is contrary to the opinion commonly held on this subject—H.S.R.

Rail End Welding

Rail end batter has been a serious problem to maintenance of way engineers for many years. Until recently, there was not much that could be done about it without removing the rails from the track. The magnitude of the problem may be visualized easily when it is recalled that there are some 280,000 miles of first main track in the United States to maintain and that good riding track is out of the question when the rail ends are allowed to become badly battered. In fact, when rail batter reaches 3/64 in., the condition is readily felt in riding over the track. The maintenance of such track requires increasingly heavy expenditures and constant supervision to keep it from becoming entirely unfit for high-speed service.

In the past, battered rail had to be replaced with new rail and the salvaged rail used on branch lines or in yards and sidings. The modern method of correcting rail end batter, however, is by welding—either by oxyacetylene or electric arc (page MA 353 L 2). By this method, the rail ends are reconditioned in the track at the small expense of $300 or less per mile, and the full length of the rail is preserved.—E.V.D.

Arsenic Poisoning

According to Chaybany (page MA 356 L 8) a cause for porosity in plated metals may be the presence of "feeble traces" of As.—H.W.G.

Elongations

Kuntze and co-workers devote 15 pages to a discussion of methods for calculated elongation in any gage length from the value for one gage length (page MA 360 L 7.)—H.W.G.

New Lattice Constants

A liberal supply of new information on lattice constants is available this month. Offner (page MA 361 R 2) has confirmed existing data on Hauerite (MnS_2). Hoffmann (page MA 361 R 2) has located the S atoms in sulphides and sulphates of As, Sb and Bi. Levi and Baroni (page MA 361 R 3) report the constants of NiS and NiSe. Neuburger, in a series of articles (page MA 361 R 4, 5, 6 and 7) reports the constants of Si, Be, Cb and Ta. Also there is announcement of Neuburger's useful annual summary of crystal structure data (page MA 361 R 8.)—J.S.M.

Magnets

Though we hear a good deal about the relation of magnetic properties and hardness, Michel (page MA 367 R 2) says there is no relation.—H.W.G.

Not Even Step-Children

In another article Michel points out another case of no relationship, this time between corrosion tests of 18:8 in pure liquids and behavior in service, in impure ones (page MA 370 R 2). Well, why go on making tests that don't correlate? It doesn't sound impossible to determine what the active impurities are and specify a suitable concentration in the rest reagent.—H.W.G.

Hard-Facing With Welded-On Overlays

Hard-facing of parts of machines and equipment with welded-on overlays is a comparatively recent development, dating back scarcely more than 15 yrs. Its original application was on oil well drills and certain types of excavating equipment, to resist abrasion and shock. As a result of the outstanding success which it achieved in these fields, hard-facing soon was introduced for many other purposes. Today, the process has already achieved considerable importance (page MA 353 L 3).

Hard-facing prolongs the life and increases the efficiency of the part to which it is applied. Not only the worn away metal of used parts may be replaced, but the loss of metal from the wearing surfaces of new parts may be minimized by the application of a thin layer of hard alloy, which protects the surface. Properly hard-faced cutting edges are self-sharpening and do not dull in service. This principle is utilized in hard-facing dredge cutter blades, power shovel teeth, road grader blades, mining bits and much other equipment with great success. The power consumption is reduced and the performance bettered thereby.

Hard-facing materials should not be used for complete build-up or replacement of metal worn away. The build-up should be made with high carbon steel welding rod. When rebuilt almost to size and shape, a comparatively thin layer of hard-facing material is applied to bring the part to finished dimensions. Enough excess is deposited to allow for grinding, where this is to follow.—E.V.D.

Things Equal to the Same Thing

Bancroft and Porter cite evidence that the film on passive iron is an oxide no higher than FeO_3 and no lower than FeO_2, hence, by gum, it is FeO_3 (page MA 370 R 4).—H.W.G.

Wear

Meyer (page MA 370 R 5) sets, as the chief criterion for a wear test, that the test must produce the same type of wear that occurs in the service for which materials are being evaluated. Elementary common sense often forgotten—H.W.G.

Bumpless Bumpers

Bumpers for automobiles that don't bump the occupants are said by Schnorrenberg (page MA 375 R 2) to be obtained by the use of Al. Other low modulus materials should act similarly.—H.W.G.

Candid Micrographs

Rossow suggests making micrographs on fine grained movie film with a special small camera (page MA 361 R 9). Some laboratories are doing this as a regular practice at quite a saving.—H.W.G.

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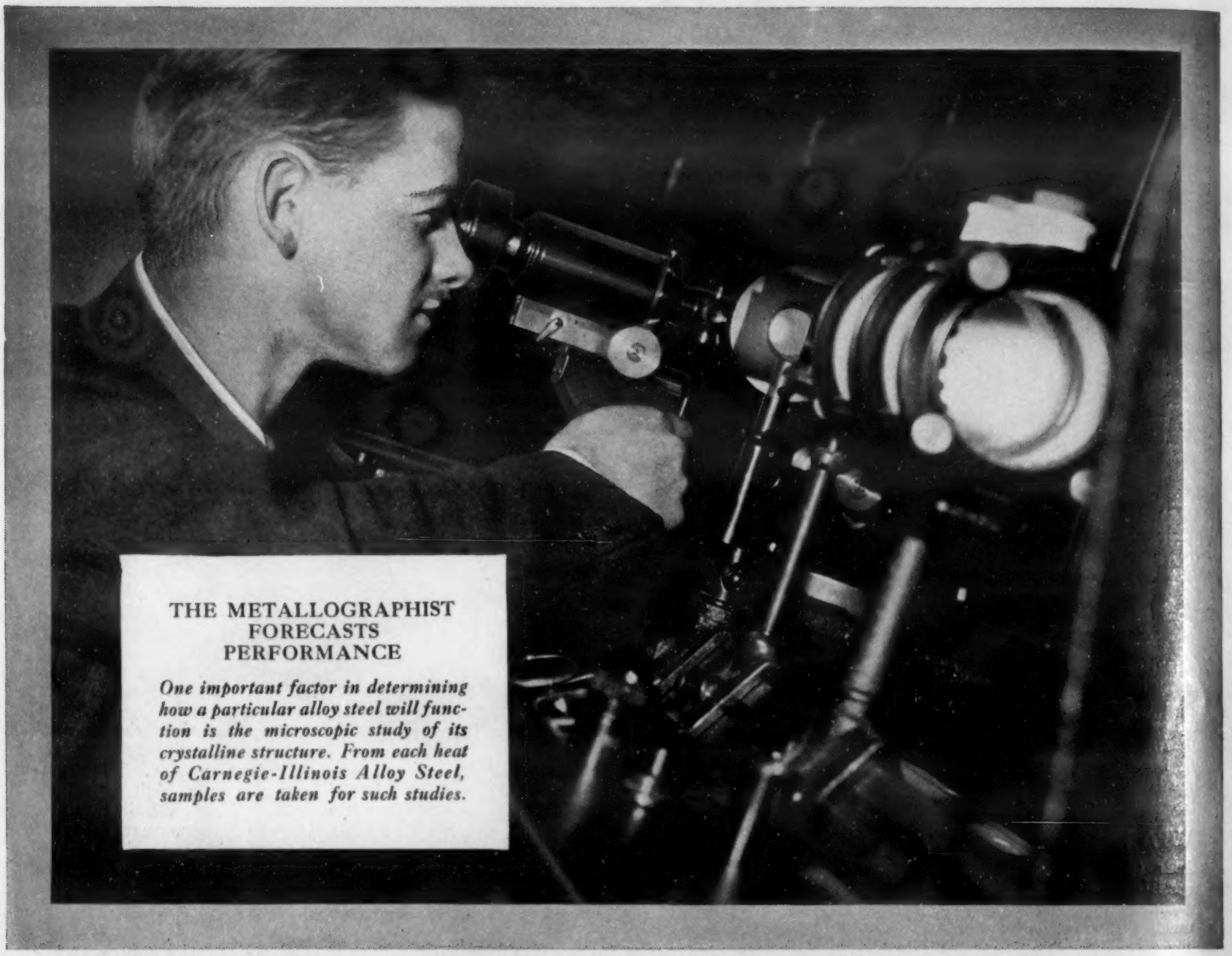
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FORECASTS
PERFORMANCE
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Our Section Editors

MAKING abstracts to give real information for engineers is not satisfactorily done by clerks. It has to be done by people who know from their own experience what the author is talking about. It is even more important that abstracts made by metallurgists for metallurgists be reviewed by still other metallurgists of wide experience and knowledge of what is going on in the particular branches of the industry that are concerned. Without such review, well known facts would be reiterated unnecessarily and new facts of vital import might not be sufficiently recognized and properly featured.

In the Section Editors who thus review the abstracts, we have a group of experienced men of broad vision. The reader sees the results of their work but probably seldom appreciates how much more useful and reliable the abstracts are because of that work. The Section Editors' initials are familiar through the "High-lights." In order that their faces may also become familiar, we are running this series of sketches about these men whose cooperation is so vital to the usefulness of the abstract section.

CHARLES S. BARRETT is engaged in research at the Metals Research Laboratory of the Carnegie Institute of Technology, Pittsburgh. He is also a lecturer in the metallurgical department. The University of South Dakota conferred on him his B. S. degree in 1925. He obtained his Ph. D. degree in 1928 from the University of Chicago. In his last year there he was awarded the Charles A. Coffin fellowship to pursue studies on the scattering and polarization of X-rays. After being graduated, Dr. Barrett spent four years in the division of physical metallurgy at the Naval Research Laboratory; he then accepted his present position.

CHARLES S. BARRETT

Dr. Barrett's interest has been chiefly in the application of X-rays to metallurgical problems. At the Naval Research Laboratory he collaborated in the development of radiography by gamma rays and in various papers on the crystal structure and microstructure of alloys. At Carnegie his work has been along similar lines, including also researches on the application of X-rays to internal stresses, cold work and fatigue.

The Institute of Metals Division of the Mining Engineers bestowed on Dr. Barrett its annual award in 1934. He is a member of several technical societies and is active in committee work.

GEORGE L. CRAIG is research engineer for the Calumet & Hecla Consolidated Copper Co., New York City.

Mr. Craig was graduated from West Virginia University in 1927 when he received the degree of B. A. He majored in chemistry. During 1927-1928 he was chemist for the Fairmont Aluminum Co., Fairmont, W. Va. He became technical supervisor of this company in 1929, serving through 1930. In 1931 he joined the metallurgical staff of the Battelle Memorial Institute, Columbus, Ohio, where he remained until 1934 when he took up his present connection.

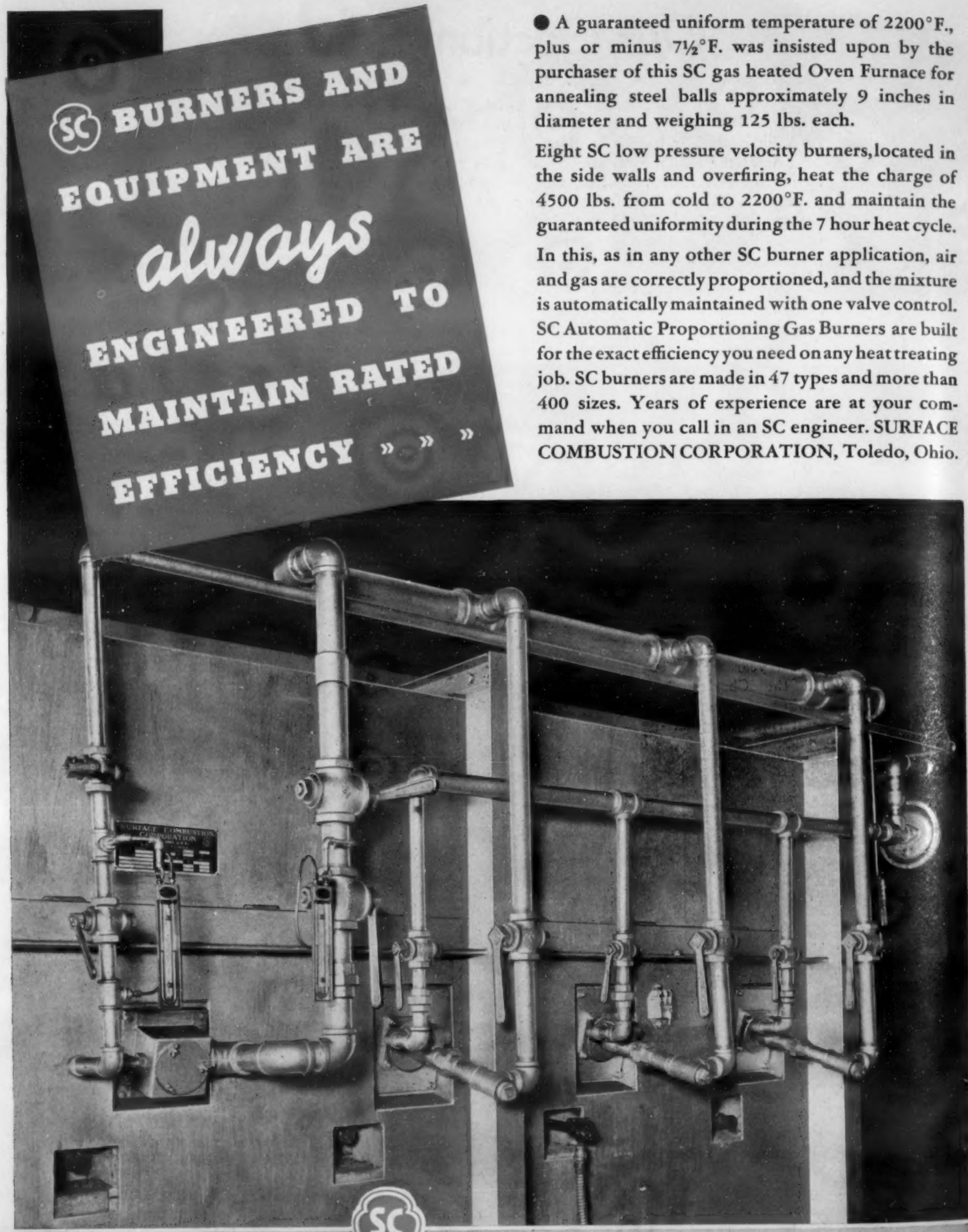
GEORGE L. CRAIG

In 1929 he received his M. S. degree in metallurgical engineering from the University of Utah.

JOHN G. ATTWOOD received his B. S. degree in chemical engineering from the Armour Institute of Technology in Chicago in 1929. Since his graduation he has been employed as a concentration engineer on the staff of the Battelle Memorial Institute, Columbus, Ohio.

JOHN G. ATTWOOD

His work has been diversified and has included research in microscopic mineralogy, fine-size float-and-sink, oxide flotation, magnetic and di-electric separation, and all phases of gravity concentration on both laboratory and plant equipment.



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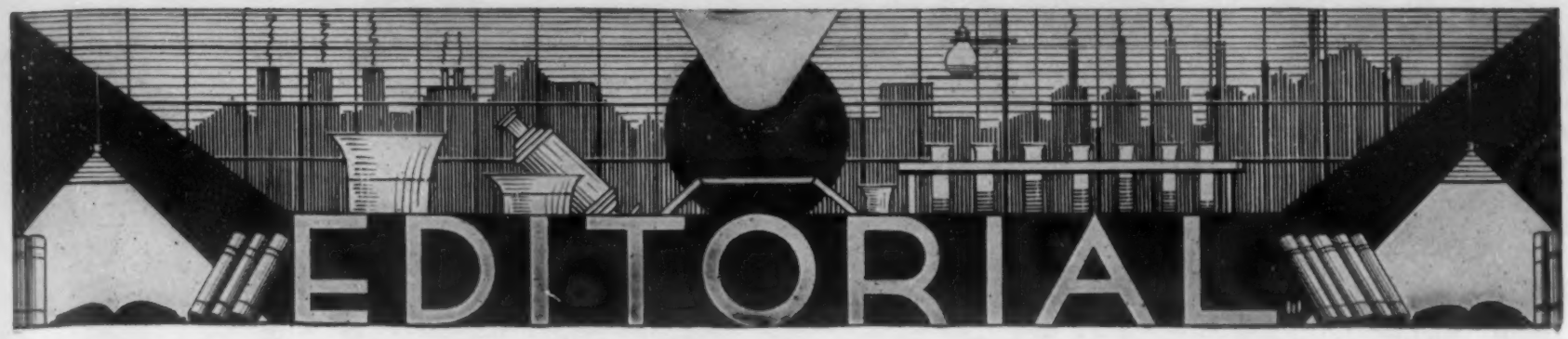
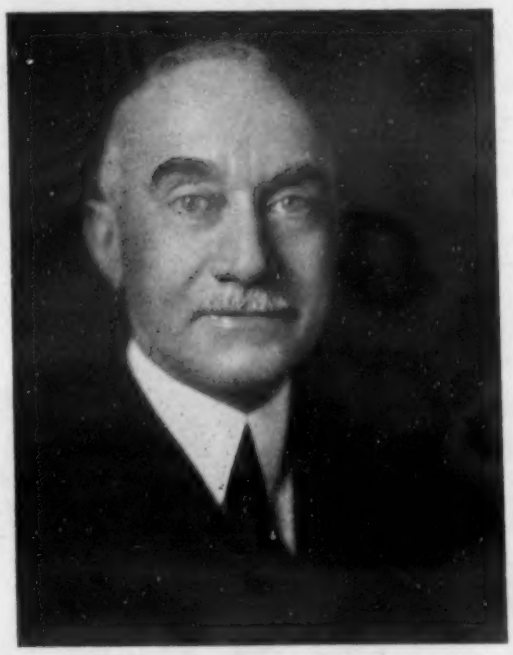


Dr. Clamer Joins Our Advisory Board

DR. G. H. CLAMER, president and general manager of the Ajax Metal Co., has accepted the invitation to join the Editorial Advisory Board of METALS & ALLOYS. Dr. Clamer is one of the pioneers of American metallurgy. Trained as a chemist at the University of Pennsylvania, he became interested in metallurgy, some of his early work having to do with copper-nickel steels, of a type that has only recently found commercial exploitation, though he clearly showed their good properties.

There was no such delay in the application of his work in the non-ferrous field with which he has become so thoroughly identified, since the success of the Ajax Metal Co. in supplying ingot metal of specified composition and purity rests very largely on his work, both as a technician and as an executive. Not content with this achievement, he became interested in the possibilities of electric melting of copper-base alloys and his foresight, patience, willingness to invest in research, and his personal encouragement of the research workers through long years of hard sledding were crowned with success in the development of the Ajax Wyatt and Ajax Northrup furnaces. The former is doing almost all of the melting in the wrought brass industry in the U. S. and a large proportion of it abroad, while the latter is commercially serving not only the non-ferrous industry but the steel industry in a very important way.



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DR. G. H. CLAMER

The value of the high frequency furnace is even greater from the scientific than from the directly commercial angle, for as a research tool it has made it easy to carry out small scale studies that it would be inconvenient, if not impractical, to carry out without it. Most metallurgical laboratories would feel that they were back in the horse and buggy days for certain, if their Ajax Northrups were taken away from them. Without detracting from the genius of Wyatt, Northrup and their collaborators we can very properly ascribe the present status of these furnaces to the technical faith and executive ability of Dr. Clamer. How modest he is in giving the lion's share of the credit to those who work with him will be evident by his account of the development of the Ajax Northrup in the May, 1935, issue of METALS & ALLOYS.

Despite the pressure of his executive duties, Dr. Clamer is too much of a metallurgist and too thorough a scientist to lose touch with the technical aspects of his industry and his constructive work along technical lines has not been interrupted. He has been the guiding spirit in the move toward simplification and standardization of copper alloys for sand castings which is doing away with the costly and troublesome practice of having a greater multiplicity of commercial alloys than is really justified.

He has always been active in metallurgical societies, his memberships in the Mining Engineers, the A.S.T.M. and The Electrochemical Society dating back to 1902. He was a charter member of the last named, and likewise an original member of the British Institute of Metals. He has been president of the A.S.T.M. and of the Am. Inst. of Metals, and has served on numerous committees of these and of the A.F.A.

Among the honors received are the honorary D.Sc. from Ursinus, the Seaman medal of the A.F.A. and the Elliott Cresson Medal of the Franklin Institute.

Dr. Clamer has long been interested in what we are trying to do in METALS & ALLOYS and has been helpful in many ways. That we are now free to call upon him, as a member of the Editorial Advisory Board, for still more constant advice and counsel gives us deep satisfaction.—H. W. G.

The Glorious Fourth

ON the 4th of July we, as a nation, have long been accustomed to having a feeling of thanksgiving for political and religious freedom, for preservation of the rights of person, of property, of free speech, and for having "no taxation without representation". Since the days of dictatorships abroad we have had added thankfulness that no individual or little group can impose his or their will on the rest of us by physical fear. The minority party is not yet subject to wholesale assassination or fear of it, and we can and do say what we think about the officials in power up to and including the Chief Executive.

In spite of the spending of our taxes on riotous Passamaquoddy's and the like without due process of law, in spite of the viciousness of Farleyism and the asininity of Tugwellism, in spite of all the attempts to steer us toward dictatorship via "must" legislation,

(Continued on page 174)

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Magnaflux Inspection Methods

In Airplane Engines

By H. J. NOBLE

Assistant to H. J. Fischbeck, chief metallurgist,
Pratt & Whitney Aircraft Division,
United Aircraft Mfg. Corp., East Hartford, Conn.

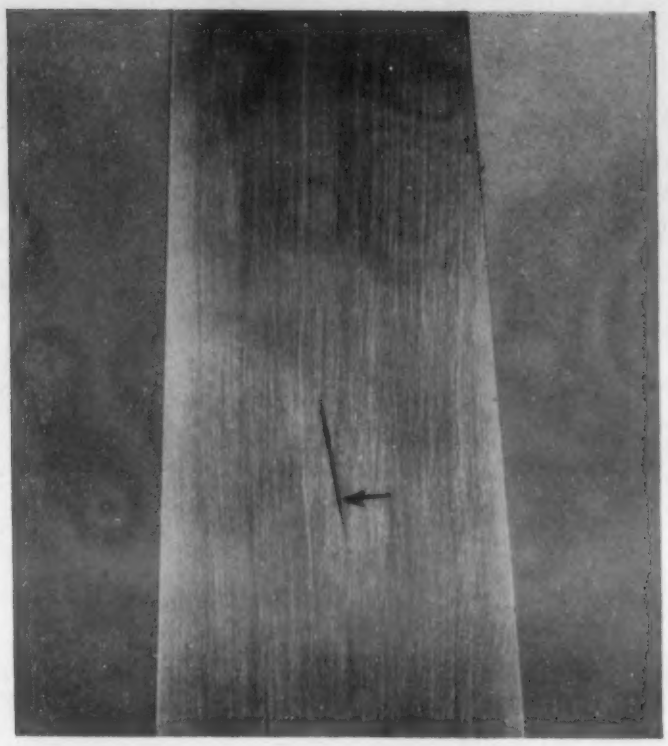


Fig. 1. Defect on a Master Rod, Located with Dry Powder.

AFTER FIVE YEARS of varying applications of the "Magnaflux" method of inspection in the aircraft field, it is interesting to note the improvements and refinements in the use of this method, its ever-increasing application in this and other industries, and its effect on both the manufacturer and the consumer of magnetic materials. Also it might be well to consider some of the peculiarities and limitations that have been encountered.

Basically, Magnaflux is a process of locating defects in magnetized steel or its magnetic alloys by means of a finely divided paramagnetic material which was developed by A. V. DeForest. Defects may consist of seams, pipes, etc., either at or below the surface, quenching cracks, grinding cracks, fatigue cracks, or faulty welds. The paramagnetic material may be applied by dusting from a shaker directly onto the work —the dry method—or by dispersion in a vehicle such as kerosene and immersing the work in it—the wet method.

Early Equipment Not Elaborate

Our first installation consisted of a single-purpose piece of equipment designed solely for the inspection of propeller hubs. It proved to be very satisfactory for that particular job. The magnetizing unit consisted of two storage batteries in parallel with no rheostat for controlling the current. Experimentation with other parts indicated that similar success could be expected, provided suitable apparatus was available. This additional apparatus would necessarily include contacts capable of accommodating pieces of various sizes and shapes, and a rheostat for current control.

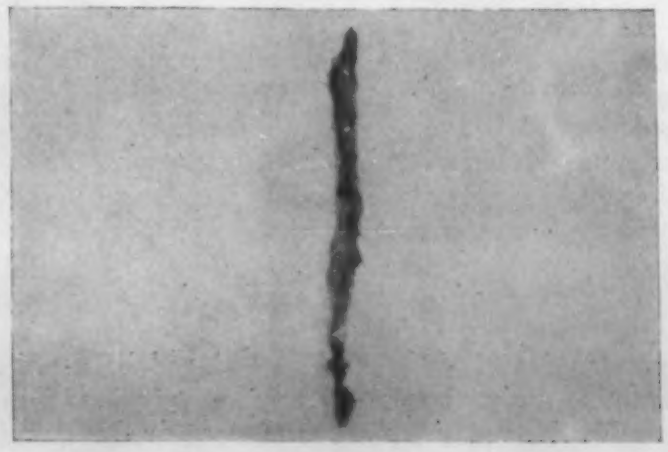
Such equipment was built under the direction of A. V. DeForest, and was similar to the now standard model A-300. The magnetizing unit in this equipment was four storage batteries, making possible a higher amperage with less attention being required to keep the batteries fully charged. About a year ago an alternating current (A. C.) magnetizing unit was added to the equipment in addition to the direct current (D. C.) derived from the batteries. The A. C. gives an amperage in excess of 3000, which is found more satisfactory on large masses such as crankshafts. The D. C. is still used in many applications, especially on the smaller pieces. More recently, several small self-contained units, (model B-203-A), have been installed at different points in the plant for routine inspection of small parts such as piston pins, knuckle pins, tappet rollers, etc.

The Dry Method

All of our first work with Magnaflux dealt only with the dry powder. This consisted of pure, finely divided iron powder, coated partly to create greater permeability which would tend to make more powder stick to a defect, and hence better visibility, and partly for the color, which also was an aid to visibility. As stated before, after the piece had been magnetized, the powder was dusted over the piece from a shaker, the excess powder removed by tapping, tilting or blowing —the powder adhered only to the defects. However, if the piece were not perfectly clean and free from all traces of grease, the powder would stick to the dirty spots and cover any possible defect. Consequently great care had to be exercised in cleaning all parts to be inspected.

In our plant, the plating room adjoins the hardening room. We found it convenient to locate our Magnafluxing equipment in the hardening room, so that the

Fig. 2. Cross Section of a Defect Shown in Fig. 1 at the Arrow. Magnification 100 diameters.



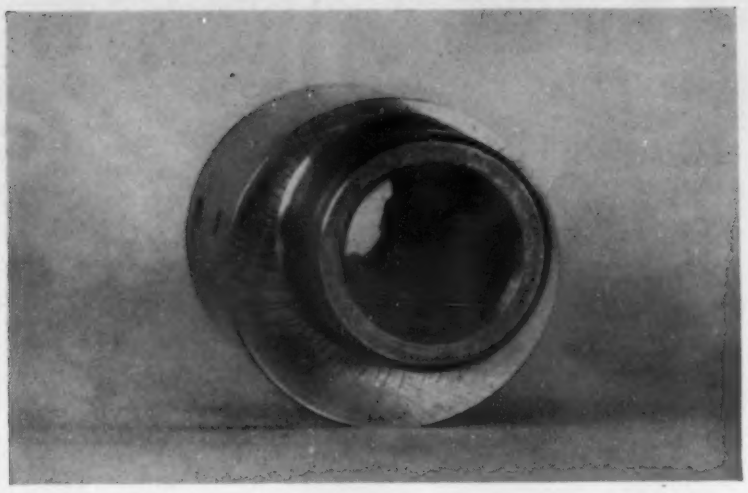


Fig. 3. Grinding Cracks in a Camshaft Detected by the Wet Method.

parts could be cleaned in the same tanks and in more or less the same manner as parts to be plated. Parts inspected after heat treating were sandblasted without going through the cleaning process. It should be mentioned here that finished parts so cleaned should be slushed as soon as possible after Magnaflux, as the chemically clean surface is apt to corrode quite rapidly, especially in sultry weather.

The Wet Method

Some time later, the wet method of Magnaflux was introduced, which we now use almost exclusively though there are some instances where the dry powder is used to advantage. The powder used in the wet method is magnetic oxide, and much finer than that of the dry method, the particle size being about five microns. This is in suspension in "Varsol," a vehicle similar to kerosene, and kept dispersed by air bubbling through small holes in pipes along the bottom of the tank. Experimentally, we tried carbon tetrachloride as a vehicle, and a tank of about 10 gallons capacity was used. This worked very well, since the parts did not have to be degreased, carbon tetrachloride being a good solvent. Also the tetrachloride evaporated quickly, leaving the parts dry. Unfortunately, the cost of such a large quantity in the proposed installation, along with high evaporation losses, made it advisable to use a cheaper medium.

Wet and Dry Methods Compared

The wet method is used in preference to the dry for several reasons. It is more sensitive and more easily discernible on a machined or ground surface. In most cases, the need for cleaning the work is eliminated. Also the inspectors have found it more agreeable to work with, as the dry powder has a tendency to be dusty at times. In the matter of sensitivity, we have found that the wet powder will adhere to cracks so small that they can be seen only under fairly high magnification, (20x binoculars are frequently used in this connection). On one occasion when we had reason to suspect the presence of a fatigue crack in a certain location, we found one less than 0.010 in. long, which defied detection at 100 diameters when the powder had been removed. Of course, the dry powder would not show anything in this case, as the particle size is too large. On the other hand, the wet powder will not pile up to give an indication of depth, regardless of the size of the defect.

In the case of a large crack with an appreciable width, the wet powder will fill up the crack and not pile up, and a black line will still be visible, even after attempts to wipe it off. If the dry powder were used, it would pile up, the deeper the crack the greater the amount of powder it would hold, and the more the powder would resist dislodgment by tapping or blowing. Thus, with a certain amount of experience, an inspector can estimate the seriousness of a defect. On rough forgings and welds, only the dry powder can be used to advantage.

Magnetizing the Sample

The most important phase of the whole inspection method is in the magnetization of the piece. For longitudinal defects, it is necessary to pass the current through the piece in such a manner that the magnetic field created will be at right angles to the defect. Theoretically and actually, if the defect is parallel to the flux, no indication of its presence can be detected. As the angularity of the defect with the flux increases, from 0 to 90 deg., the presence of the defect is more strongly indicated, with 90 deg. being the maximum. For that reason, irregularly shaped parts must sometimes be magnetized in as many as half a dozen different directions. That means that the pieces are magnetized in one direction, inspected by wet or dry powder, demagnetized, and the process repeated until all possibilities are covered. We found that in magnetizing the piece in two directions at one time, the magnetization in the one direction was so diminished by the other magnetization that we could not be sure of the results. Consequently, each magnetization now is a complete operation.

Although an electrician is by no means needed for this work, a few of the underlying principles of electricity and magnetism are very useful, especially to the one in charge of the installation. Probably almost as important is knowledge and experience in the type of defect that might be expected, and where located. For example, if a simple piece, such as a bolt, were made from heat-treated bar stock, the only type of defect we might expect would be longitudinal seams, etc., which would require but one magnetization. If it were made of annealed stock, and heat-treated after rough machining, the possibility of a crack under the head arises, so that magnetization in two directions is necessary. Assuming a third possibility, that of the piece having been in service for some time, and being back for reinspection, we could safely assume that there were no longitudinal defects, or that they were inconsequential, and we would magnetize in a way to show up any possible fatigue cracks, either under the head or in the threads. An experienced man is therefore necessary in setting up the procedures to be followed, both at the time of initial installation, and when new or complicated parts are added to this routine.

Overcoming Magnetizing Difficulties

A few special cases, where difficulty in magnetizing was experienced, will be described. In magnetizing

Fig. 4. Crack at the Root of a Tooth in a Cam Rim.

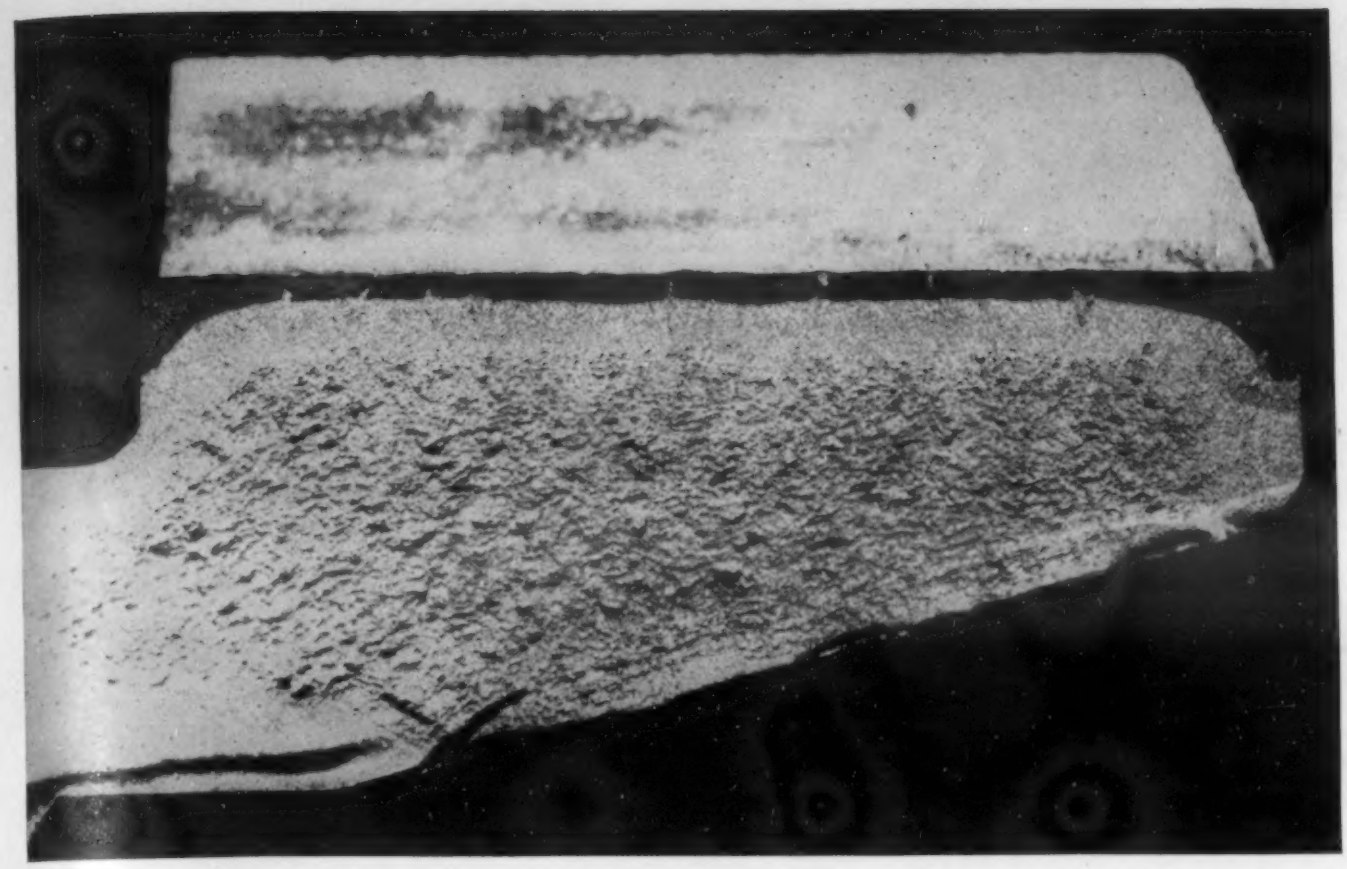


Fig. 5. Face of the Crack in Fig. 4, Showing No Fatigue after 1000 hrs. Service. Reduced about 60 per cent from an original magnification of 10 diameters.

large crankshafts, a copper bar was placed through the hole in the crank pin, and again through the main shaft with the current going through the bar to induce magnetism. This method eliminated any possibility of burning at the points of contact, but the induced magnetism was not so great as that produced by the current passing through the shaft directly. To assure good contact, large copper balls were placed against the holes. Along this same line we use flexible battery connections against irregularly shaped pieces, which gives a greater and more uniform contact.

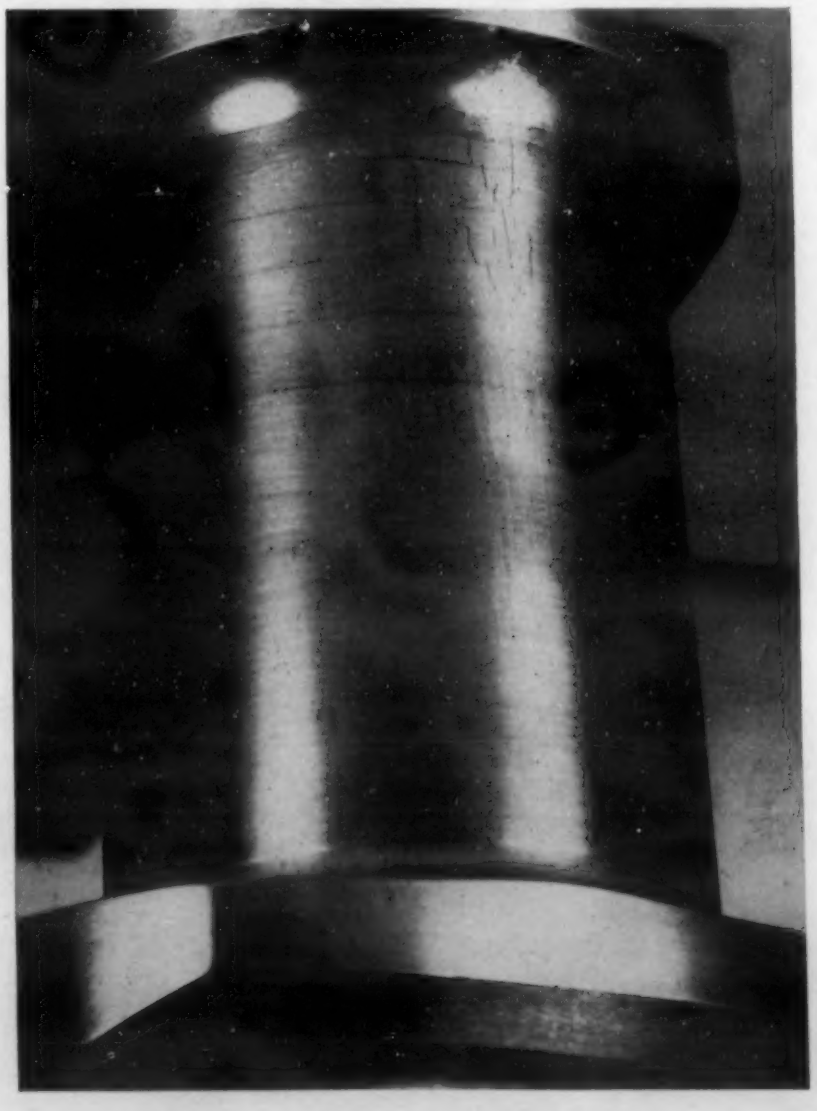
The magnetization of cams requires special technique. A cam, as usually employed in a radial engine, is a rim with external lobes, and gear teeth either internal or external, about 8 to 10 in. in diameter. We could not pass the current through the piece flatwise, which would produce a flux in the desired direction, because the face plates on the Magnaflux equipment are not large enough. Induced magnetism was resorted to by placing a copper bar within the cam rim between the plates of the magnetizing unit. If the bar were at the center of the cam rim, the magnetic induction at that distance would be negligible. The cam was moved in a circular motion around the bar, while the direct current was on, so that the whole cam was brought in close proximity to the bar. Obviously, A. C. could not be used in this instance.

Some of the Defects Found

At the time we started using Magnaflux, this method of inspection was in its infancy, and accurate data were still being gathered on the types and characteristics of defects which might be detected in this way. We believed the method offered great possibilities, and to further the benefits of more accurate inspection, we spent considerable time in studying and analyzing rejections. In some cases, the defect could be seen with the naked eye, once the Magnaflux had located it, while in others it could not. At other times, we found visual defects which would hold no powder. Enough samples of different types of defects were examined in the laboratory to enable us to interpret more intelligently the story that Magnaflux was trying to tell. These experimental pieces were broken open, cross-sectioned, examined microscopically, deep etched, or handled in the manner that would yield the most information.

Fig. 1 shows one of the early defects found on a master rod by dry powder. The defect was visible to the eye only a small part of the actual length. We also noticed that one end of the powder pattern appeared to be very fuzzy, and although the powder would stick to it, slight blowing would start to dislodge

Fig. 6. Defects in Crank Pin of a Crankshaft.



it at the end. Approaching the other end, it became impossible to blow the powder off. We found in our laboratory examination that this was an oxide inclusion which ran at a slight angle to the surface. Fig. 2 shows a cross-section, (100 diameters), above the point which resisted being dislodged by blowing, and is located by the arrow in Fig. 1. Other defects, which were discernible to the eye, (especially on pieces made from bar stock) but which would not show up with Magnaflux, were proved to be surface defects or hair-line seams, with a width greater than depth.

Grinding cracks are a rarity now, because of the careful check on parts where they might occur. Fig. 3 is a pinion shaft, which is ground on the smaller outside diameter and ends of the large diameter at the same time. The side of the grinding wheel does not produce so good a cutting medium and becomes rather easily loaded. Care must be exercised to prevent the condition illustrated here.

Fig. 4 shows a section of a cam returned by a customer after 1000 hrs. of service. This crack was located with Magnaflux during a major overhaul. The powder had been covered with lacquer and sent in to our service department so that their reason for returning it was still apparent. The piece was broken open for examination of the faces of the crack as shown in Fig. 5. We concluded that this crack had occurred in straightening, an operation necessary in cases where the cam goes out of round beyond our limits in heat treating. In other words, the crack was present at the time of manufacture, but had escaped detection by our inspection department, as Magnaflux was not used on this part at that time. Furthermore, it had been subjected to several periodic inspections by the customer. After Magnaflux equipment was installed by the customer, the defect became quite apparent. Fortunately, the crack had not progressed any during operation, as there was absolutely no indication of fatigue. Although we would not knowingly pass any part with such a defect, we were glad to get it back with its history because it added to our general knowl-

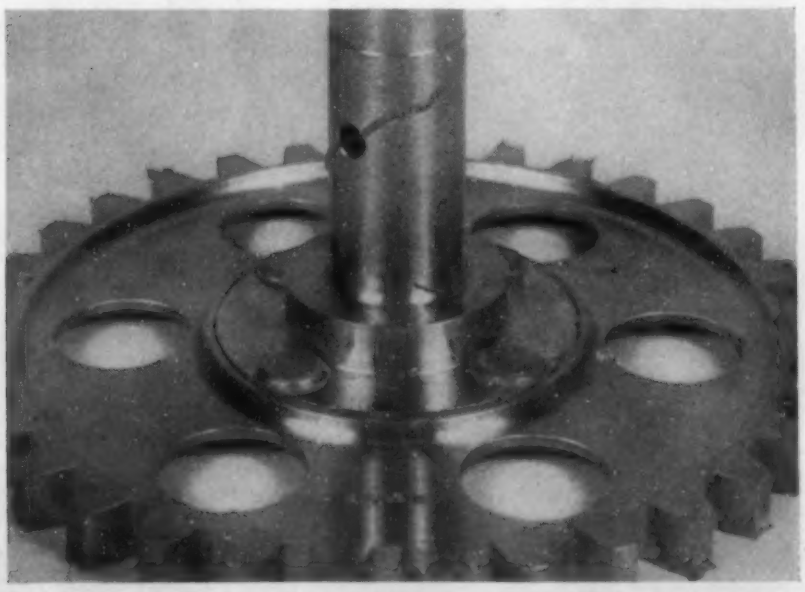


Fig. 7. Fatigue Crack in a Gear Shaft Indicating the Way the Dry Powder Will Pile Up on a Deep Crack.

edge of the effects of various kinds of defects with respect to location and stress concentration.

Experience with Crankshafts

Crankshafts are purchased by us in a semi-finished condition. They are magnafluxed when received, before any further machining is done. Fig. 6 is one of the rejections, which represents one of the worst we have seen as to size and number of defects. Figs. 7 and 8 represent fatigue cracks located in the one case by dry powder, and in the other by wet. These two samples were found after bench tests of experi-

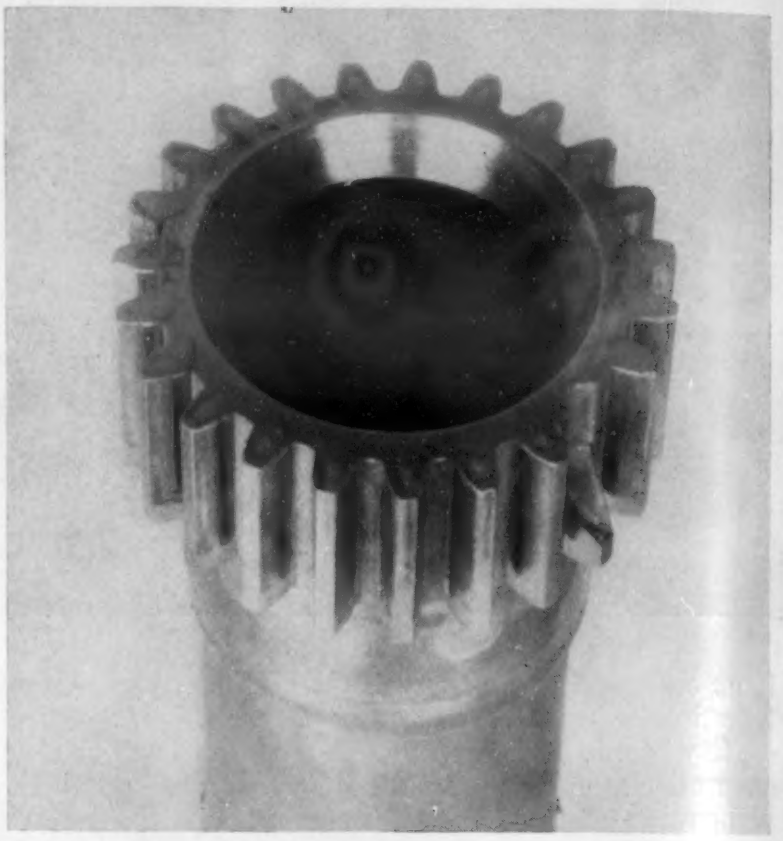


Fig. 8. Fatigue Cracks at the Root of Teeth in a Gear Located by the Wet Method.

mental engines. The spiral crack in the shaft of Fig. 7 was just about visible to the eye without the powder, but there was no room for doubt after the use of Magnaflux. Although one tooth was broken out of the gear in Fig. 8, and one might expect other teeth to be similarly cracked, only two were identified before the Magnaflux wet method was used, while 11 were found afterward. This is an indication of the value of this method of inspection, especially in experimental work where new design is frequently employed.

Because of its inherent characteristic as a non-destructive, as well as a quite rapid test, Magnaflux has become almost indispensable. Countless parts can be checked where otherwise a representative part would have to be tested to destruction. A safer and more uniform product is therefore guaranteed. As far as the steel makers are concerned, it has been and will continue to be somewhat of a hardship to fabricate material which will pass this test. However, we have found them quite willing to cooperate, and earnest efforts are constantly being made to improve the quality of their product. During the period we have used the Magnaflux test, we have noticed a gradual decrease in rejections due to metallurgical defects. Also defects produced mechanically, such as by grinding or heat treating, are fewer, due to the closer surveillance over manufacturing operations, whereby any trouble is quickly identified and corrected.

The hardness tester has won a definite place in inspection procedure. Although Magnaflux is in an entirely different field, we believe it is rapidly approaching the hardness tester in importance, and that time will see its acceptance as such in all quality production.

Residual Metals in Open-Hearth Steel

Arsenic, Antimony and Others

By JOHN D. SULLIVAN AND RAY A. WITSCHEY

Battelle Memorial Institute, Columbus, Ohio

THIS IS THE FIFTH of an annual series of articles on residual metals in open-hearth steel. Measurable amounts of copper, nickel, chromium, tin, arsenic, and antimony are found in practically all open-hearth steels. Lead, zinc, molybdenum, and vanadium are also present in smaller amounts. Residual manganese, silicon, and aluminum are found in steels, but since they are intentionally added to the charge they should not be classed as truly residual constituents. Copper is present in some of the iron ores of the United States, and is found in pig iron made from such ores. Copper also enters steel through scrap such as bushings, wire, tubing, and from copper-bearing steel and iron scrap.

Most of the residual nickel probably comes from iron and steel scrap although a few foreign ores contain considerable nickel. Chromium comes largely from alloy steel scrap, but some iron ores contain appreciable amounts of this element. Tin comes from such sources as de-tinned scrap, cans, bearings, and galvanized scrap. Arsenic is found in small amounts in many iron ores and in larger quantities in some of the Southern United States and foreign ores. Antimony probably comes partly from non-ferrous alloys such as bushings and bearings that accompany ferrous scrap.

Virtually all of the copper and nickel in the blast furnace and open-hearth charges is recovered in the metal. In the open-hearth the recovery of chromium depends on oxidizing conditions, length of the refining period, slag composition, and carbon content of the steel. Probably nearly all the tin and a high percentage of both arsenic and antimony remains in the steel. Zinc is largely eliminated by volatilization and part of the lead volatilizes and part of it settles through the bath into or through the furnace bottom. Molybdenum is nearly all recovered but is present in small amounts. Vanadium remains in the steel to a large extent although partly as oxide.

Previous Records

In previous articles[1] analyses were presented on the content of certain residual metals in samples submitted by various steel companies in the United States and Canada. Samples are taken from representative heats before alloy additions are made. At the end of the month composite samples are made and sent to Battelle Memorial Institute. Here, annual composite samples are made for analysis. During 1935, 18 companies representing an annual tonnage capacity of approximately 8,000,000 tons cooperated. This study continues from year to year, and this report brings the results up to date.

Different plants may take samples at different stages between the time of melt-down and the time for addition of ferro alloys, and the differences in sampling procedure may affect the figures for oxidizable elements to some degree but on the whole the data indicate the composition of the "plain-carbon steel" base used in the various plants before adjustment of

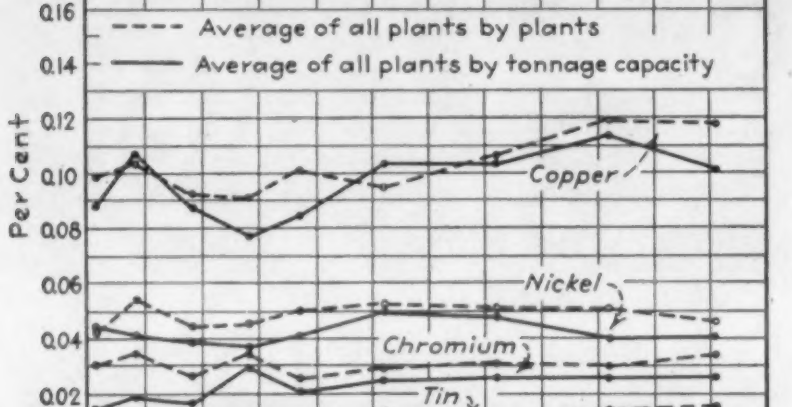


Fig. 1. Composition Averages of All Plants for the Nine Periods.

the composition by alloying elements. The residual metals in the samples, therefore, represent those coming from the scrap, pig iron, and ore.

Residual Metals in Steel

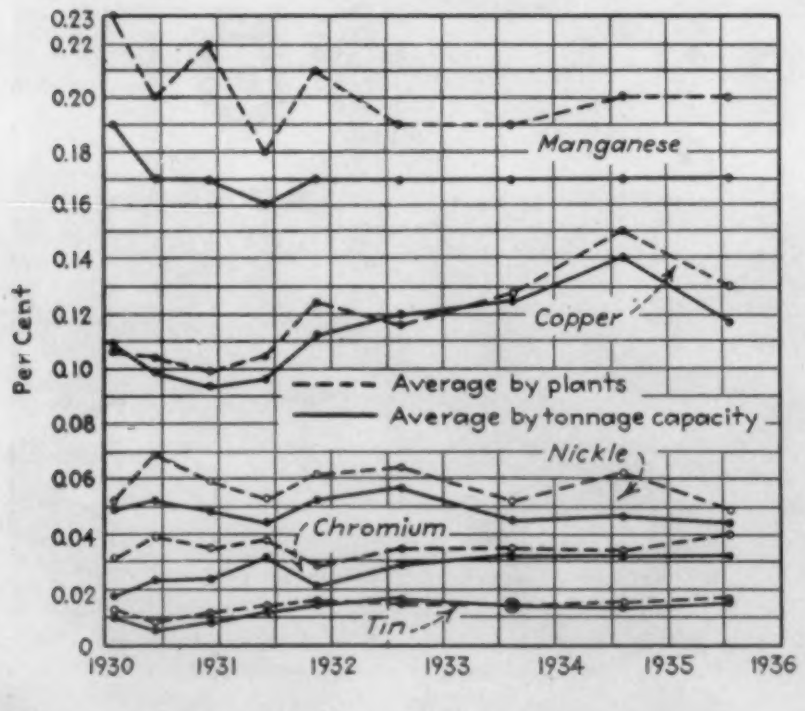
This year, because of added interest in the steel industry, results are given for arsenic and antimony. Figs. 1 and 2 contain the results of the analyses for manganese, copper, nickel, chromium, and tin for the nine periods covered in this study. The average of all plants for any period refers to those plants that cooperated in that period. The year is plotted as the middle of the interval represented.

In comparing average values of residual metals for the current period with the previous period, the following will be seen. The average of all plants, by tonnage capacity, shows nickel and chromium remaining constant, a slight increase in tin, a decrease of two points in manganese, and an appreciable drop in copper. The average, by plants, shows an increase of one point in manganese, a slight increase in tin, a noticeable increase in chromium, a noticeable decrease in nickel, and an appreciable drop in copper.

The average, by tonnage capacity, of the 10 plants that have cooperated in all nine periods shows manganese remaining constant, a slight increase in tin and chromium, a slight drop in nickel, and an appreciable drop in copper. In the same 10 plants the average, by plants, shows manganese constant, a slight increase in tin, an appreciable increase in chromium, and appreciable drops in both nickel and copper.

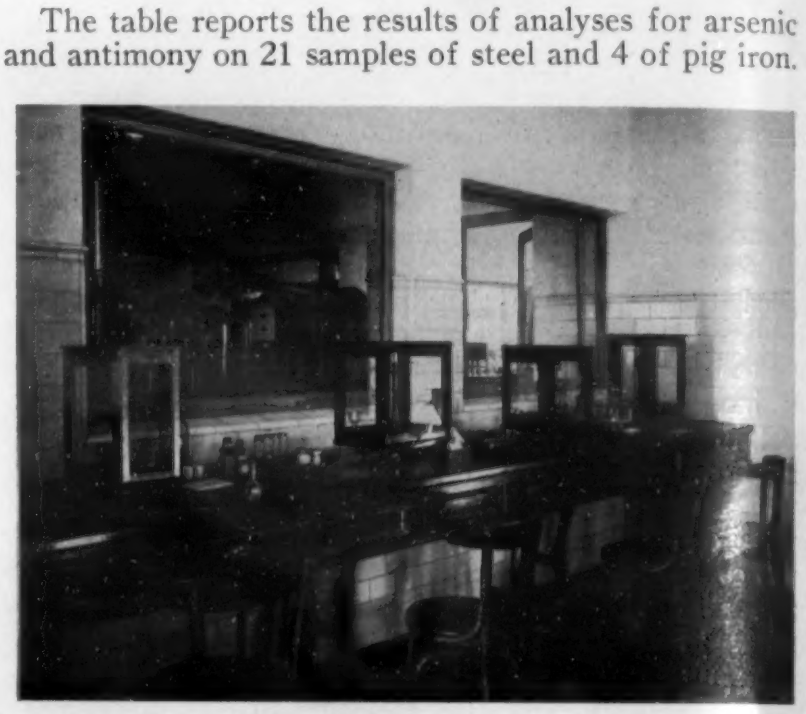
The most outstanding change is the decrease in copper content. During the eighth period the increase in copper over that of the seventh period was material, but the drop in the ninth period puts the content back to about the same place it was two years ago. The slight but persistent increase in tin and the drop in nickel are also worthy of mention. Lead and zinc were determined on several samples but in each intance the amount present was less than 0.001 per cent.

Fig. 2. Composition Averages of Ten Plants that Have Cooperated in All the Periods.



Arsenic and Antimony

The table reports the results of analyses for arsenic and antimony on 21 samples of steel and 4 of pig iron.



The first 18 samples in the table are those used in the determinations shown in Fig. 1.

Table of Percentages of Arsenic and Antimony in Plain Carbon Steel

Plant	Arsenic, Per cent	Antimony, Per cent	Sample
35-19	0.011	0.012	Steel
35-20	0.002	0.006	Steel
35-21	0.034	0.010	Steel
35-22	0.009	0.007	Steel
35-23	0.013	0.008	Steel
35-25	0.007	0.007	Steel
35-26	0.006	0.008	Steel
35-27	0.007	0.004	Steel
35-28	0.009	0.010	Steel
35-29	0.013	0.014	Steel
35-30	0.006	0.009	Steel
35-31	0.009	0.006	Steel
35-32	0.009	0.008	Steel
35-33	0.013	0.008	Steel
35-34	0.011	0.011	Steel
35-35	0.009	0.028	Steel
35-36	0.005	0.023	Steel
35-38	0.011	0.008	Steel
35-39	0.013	0.008	Steel
35-40	0.013	0.008	Steel
35-41	0.014	0.008	Steel
35-42	0.002	0.015	Pig Iron
35-43	0.002	0.006	Pig Iron
35-44	0.002	0.001	Pig Iron
35-46	0.007	0.004	Pig Iron
Average of 21 steel samples.	0.010	0.010	.....
Average of 4 pig iron samples	0.003	0.006	.....

Use of Scrap in Open-Hearth Furnaces

The curves in Figs. 3 and 4 have been plotted to show the variation in the amount of scrap used in the various years and to permit comparison of this with prices and production rates. Scrap percentages up to and including 1934 were obtained by calculation from statements of basic pig iron and basic open-hearth steel production contained in the annual statistical reports of the American Iron and Steel Insti-

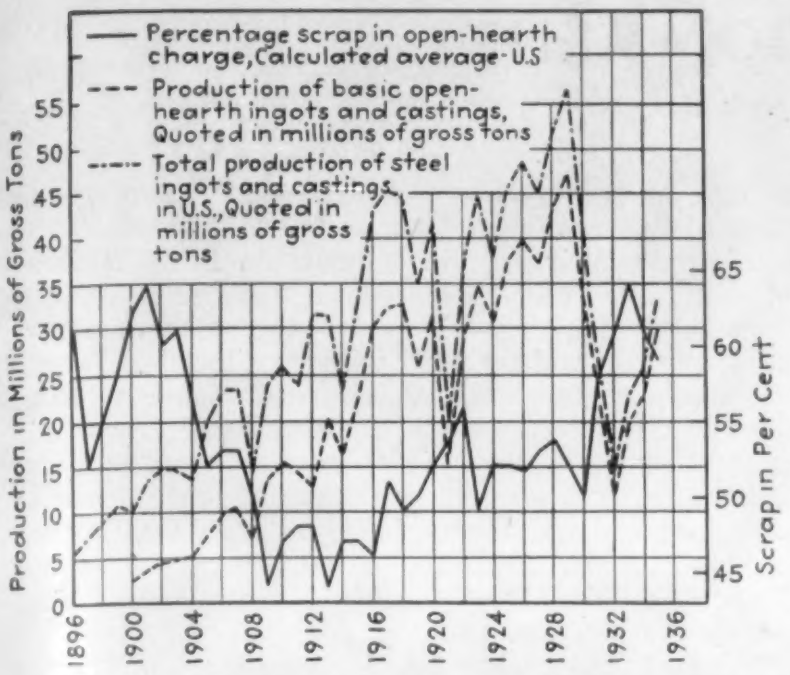


Fig. 3. Showing Relationship Between Scrap Ratio in Charge and Total Steel Production.

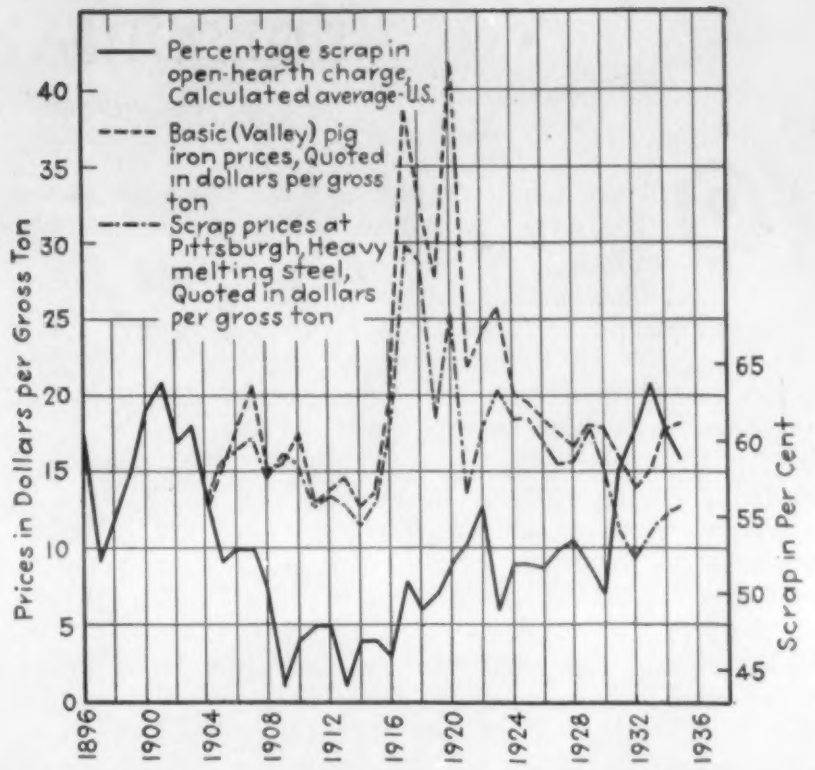


Fig. 4. Showing Relationship Between Scrap Ratio in Charge and Prices of Pig Iron and Scrap.

tute. For 1935 statistics for basic pig iron production were obtained from a statistical bulletin of the American Iron and Steel Institute, while steel ingots and castings production data were taken from *The Iron Age*. The 1935 figures may change slightly when final statistics are published.

In determining the percentage of scrap used, it was assumed that (1) an ingot recovery of 88 per cent of the metal charged was made, (2) metallic addition agents equivalent to 1 per cent of the charge were used, and (3) iron ore containing 50 per cent of recoverable iron was used to the extent of 5 per cent of the pig iron charged. As used in this report, the percentage of scrap is taken as the percentage of scrap in scrap plus pig iron. The calculated percentage of scrap used in 1935 was 59 per cent, a drop of 2 per cent from 1934, and of 5 per cent in the last two years.

Fig. 4 shows the relation between scrap in the charge and prices of pig iron and scrap. The spread in price between scrap and pig during 1935 was about $5.40 compared with about $5.30 in 1934.

References

[1] Clyde E. Williams and John D. Sullivan. "Residual Metals in Open Hearth Steel." METALS & ALLOYS, Vol. 3, 1932, page 240.

Clyde E. Williams and John D. Sullivan. "Residual Metals in Open-Hearth Steel." METALS & ALLOYS, Vol. 4, 1933, page 151.

John D. Sullivan. "Residual Metals in Open-Hearth Steel." METALS & ALLOYS, Vol. 5, 1934, page 145.

John D. Sullivan. "Residual Metals in Open-Hearth Steel." METALS & ALLOYS, Vol. 6, 1935, page 134.

Appendix — Method of Analysis for Arsenic and Antimony

IN last year's paper [METALS & ALLOYS, May, 1935] the methods of analysis used for copper, nickel, tin, and chromium were given. Since small amounts of arsenic and antimony are not usually determined in steel laboratories, the method used in this work is outlined. The reader is warned that careful attention must be given to details of the outlined procedure in order to obtain accurate results.

Arsenic and Antimony in Plain Carbon Steel

Transfer a 10-gram sample to a 600-c.c. beaker, add 100 c.c. of H_2O and 125 c.c. of HNO_3 (2:3). Cover and let stand until vigorous action ceases. Transfer to a hot plate and heat to complete decomposition of sample. Boil for 10 min. Gradually add an excess of 2.5 per cent $KMnO_4$ solution and boil for 5 min. Oxides of manganese should separate when an excess of $KMnO_4$ is present. Add sulfurous acid (saturated solution of SO_2 in H_2O) slowly until solution clears. Boil for 10 min. Cool to room temperature and dilute to 500 c.c. Pass in a rapid stream of H_2S (from tank) for 45 min. Let settle for 2 hrs., or better, overnight. Filter, on Whatman No. 2 or equivalent paper, and transfer precipitate to filter paper with a 2 to 5 per cent solution of $(NH_4)_2SO_4$ acidified slightly with H_2SO_4 and saturated with H_2S.

Place precipitate and paper in a Kjeldahl flask. Add 5 grams Na_2SO_4 and 25 c.c. concentrated H_2SO_4. Place a funnel in the mouth of the flask to cut down the loss of H_2SO_4, and digest on a Kjeldahl rack until the solution is clear and carbonaceous matter decomposed. Ordinarily this requires about 2 hrs. Cool. Dilute with 50 c.c. of H_2O. Filter into an arsenic distillation flask. (For example, see p. 375, Lundell, Hoffman and Bright, "Chemical Analysis of Iron and Steel," New York, 1931.) Wash the Kjeldahl flask and filter with 50 c.c. of HCl (1:1). Discard the filter paper. Add 75 c.c. of HCl to the distillation flask and start the distillation. Receive the distillate in a 400-c.c. beaker containing 25 c.c. of H_2O. The solution in the beaker must be kept cold. Surrounding the beaker with ice water is recommended. Distill until only 40-45 c.c. remains in the flask. Add two additional successive 25 c.c. portions of HCl through the dropping funnel and distill after each addition. Wash down the condenser with a little water.

Pass a rapid stream of H_2S through the cold distillate for 15 to 20 min. Let stand for 2 hrs. and filter through a weighed Gooch crucible. (The crucible should be brought to constant weight so that two successive weighings check to 0.2 mg. after alternate washing with 10 per cent, by volume, HCl and distilled water, and drying at 110°C. for 1½ hrs.) The precipitate is transferred and first washed with cold HCl (1:5) and then with 95 per cent ethyl alcohol. Fill the crucible nearly full of CS_2 and set it in a covered beaker for 1 hr. Wash it under suction first with CS_2 and then with 95 per cent alcohol. Dry for 1½ hrs., cool and weigh as As_2S_3. The conversion factor $2As/As_2S_3 = 0.609$.

Neutralize the residual solution from the distillation with ammonia. Add 10 c.c. of HCl and dilute to 300 c.c. Cool the solution and pass a rapid stream of H_2S for 30 min. Let stand for 2 hrs. and filter. Wash with 2 to 5 per cent $(NH_4)_2SO_4$ acidified slightly with H_2SO_4 and saturated with H_2S. Transfer the paper and precipitate to a Kjeldahl flask. Add 5 gms. Na_2SO_4 and 25 c.c. H_2SO_4. Put a funnel in the neck of the flask and heat on the Kjeldahl rack until the solution is clear and carbonaceous matter destroyed. This requires about 1 hr. Cool, transfer to a 500 c.c. Erlenmeyer flask, add 20 c.c. of HCl, dilute to 200 c.c. and cool to 10 deg. C. or lower. Titrate with $KMnO_4$ to an end-point that remains permanent for 30 secs. The $KMnO_4$ solution is made by dissolving approximately 0.57 gram $KMnO_4$ in water and diluting to 1 liter. Standardize against sodium oxalate. Fe value of $KMnO_4 \times 1.09 = Sb$ value.

A blank must be carried through all steps and the correction applied.

EDITORIAL COMMENT

(Continued from page A19)

we can still say what we like and we can vote as we like. Our ballots in November will not contain only the "Ja" column.

In other words, though they are doing the best they can, our would-be dictators are just pikers and there is still reason for thankfulness on the 4th. The rights guaranteed to us by the Constitution can still be snatched back, even though some are temporarily withheld. There is a big contrast between our condition and that of the dictator-ridden countries.

When we consider the state of fear that obtains under those dictators in regard to politics and religion and the way their vassals are ordered around by direct edict or by the artificial working up of mass psychology, it seems noteworthy that no scientific or technical overlord has arisen anywhere to attempt to influence the workers in science and technology to draw conclusions at variance with their own real beliefs or to attempt to censor their publications. Scientists are made of different stuff from dictators. The dictators can impose their will in unprovable fields such as religion, but in those fields where truth or falsity can be established by experiment, facts rather than personal opinions and personal wishes, still rule.

Before the time when laboratories became common, a scientist's beliefs were in the same category as his political or religious beliefs, as Galileo found out. Today engineering truths are supreme and no matter how much Hitler and Mussolini might like to impose their wills in scientific realms, if some one pointed out to them that they were failing to control everything, it could not be done.

We don't know what date in the history of science corresponds to the 4th of July. If it could be picked out, the scientists and engineers of the whole world might well join in celebrating it.—H.W.G.

More Money for Steel Research

RECOGNITION of the vital importance of research to the steel industry is making rapid strides. According to a survey by the American Iron and Steel Institute, expenditures for research work by 42 companies representing nearly 90 per cent of the country's steel capacity in 1936 will exceed 1929 by almost 6 per cent. The 1936 budget calls for $9,200,000. which compares with $8,100,000. in 1935 and $8,700,000. in 1929 for the same companies. The expansion this year over 1935 is also substantial. Since "money talks," this evidence is conclusive as to the place in the economics of the industry now accorded by executives of many large companies to research. A few years ago this was by no means the case—by some it was regarded as an "expensive luxury."

The direction which this research program is taking is also indicated by the institute's survey and is interesting and significant. It is stated that research this year will be directed largely toward improving the quality of the industry's products. Closely associated with this will be the finding of new markets and new uses for steel, the developing of new types of products, and the reducing of costs. It is estimated that approximately 40 per cent of this year's budget will go towards developing means of improving the quality of steel while about 20 per cent is assigned to developing new products with 12 per cent spent in finding out where and how such products can be used to the best advantage. Another 18 per cent is assigned to efforts to reduce steel manufacturing costs with about 10 per cent devoted to a study of new markets and new uses.

The fact that a large proportion of the research funds is being devoted to the improvement of products reflects the progress that is and has been made in this field. The better quality of many of the industry's products in recent years is due largely to research, induced of course by the increased severity of the demands of customers and inspection methods. And the research program in other directions redounds to the benefit of both producers and consumers.

Interesting sidelights include a statement to the effect that the facilities and equipment necessary for the steel industry's research program are valued in excess of $6,000,000. with almost 2,200 technical experts devoting their full time to this work and with about 300 other employees sharing research with other duties. As to cost, the institute estimates that research expenditure amounted in 1935 to about 37 cents on every ton of finished products of the companies included in the survey—probably not an excessive expense, certainly one fully justified by achievements both past and future.—E.F.C.

A Few Chuckles

"Insulted Wire!"

Quotation from the *Columbus Dispatch:* "The sign contains 1084 ft. of neon tubing and almost 70 miles of insulted wire, enough to run two lines from Baltimore to Washington."

Insulted wire should have been used for telegraph lines, to carry the messages to and from Washington, copied off by the Federal Communications Commission for the New Deal inquisition carried on by Senator Black.—H. W. G.

"Crystallized Metals" and Fatigue

Quotation from the *G. E. Review,* editorial, Vol. 39, 1936, page 123:

"This (the statement that metals become crystallized on fatigue) is a gross error for metals and alloys are always crystalline when in the solid state. What happened is that, under the conditions of fatigue failure, *the grains grew to readily observable size.*" (Italics ours.)

A popular misconception corrected with a worse one!—From an anonymous reader in Waterbury, Conn.

A New Metal

Quotation from Skilling's *Mining Review,* Feb. 15, 1936, p. 2:

"During 1935 a sulphur recovery plant was installed at an outlay of $2,500,000; a new industry for the production of metallic sulphur."

BERYLLIUM AND ITS ALLOYS—I

A Correlated Abstract

By JACK DELMONTE, Sc. M.

East Lansdowne, Pa.

BERYLLIUM, AS A METAL, had inspired metallurgical research at one time by offering possibilities of structures combining great strength and low weight. In that capacity the great expense of the metal was discouraging, but as an alloying medium beryllium is of immediate importance and interest. The following paper outlines the development and the production of beryllium and beryllium alloys, pointing out the difficulties that beset early investigators and the progress that has been made since then. The extent of the possible applications of beryllium alloys is emphasized, and opinions are ventured upon the future trend.

Historical

IN attempting to show similarities between the precious stones, emerald and beryl, Vauquelin in 1797 revealed a hitherto unknown constituent, common to both stones. For a while this new element was called glucinum because of its property of forming sweetish-tasting salts. As a matter of fact, this name is still retained in the French scientific circles, but it has given away in other countries to the more popular name of beryllium. For a long time, even to as late a date as 1920, it was the common belief of the scientific world that beryllium was one of the rare earth elements.

It was not until 1828, that the celebrated German scientist, Wöhler, who in the year before, first isolated aluminum from its salts, succeeded in isolating beryllium from its chloride by heating it with flat pressed balls of calcium. For almost 70 years there was no research upon beryllium that is worthy of mention, outside of random discoveries of the unusual chemical behavior of the salts of the metal. Nevertheless, in 1889, Le Beau instigated new methods of producing beryllium through the electrolysis of fused compounds. He fused a double fluoride of beryllium ($2\ NaF.BeF_2$) in a nickel crucible, inserted a carbon rod to serve as the cathode, and passed electrical current through the fused mixture. The beryllium yield, very small and with a purity of 92 per cent, was extremely hard and brittle, and in the form of steel gray, hexagonal plates. In addition to researches on the production of beryllium, Le Beau investigated several beryllium alloys, observing in particular, the very marked change in appearance of copper on the addition of as little as 0.5 per cent beryllium. Alloys with chromium, tungsten, molybdenum, and carbon were also studied.

Following the work of Le Beau, an American scientist, C. L. Parsons, attracted considerable attention by his contributions to the chemistry of beryllium[1]. In his book on the chemistry and the literature of beryllium, he discusses critically the beryllium research that had been conducted up to his time, pointing out the numerous difficulties that beset such work. In addition, he compiled a complete bibliography of all the beryllium research papers that had been published. Other all-inclusive bibliographies of the work on beryllium have appeared since that time. M. J. Meller summed the beryllium research up to 1923[2]; and Hoyt and von den Steinen did likewise in 1931[3].

Occurrence of Beryllium

Beryllium occurs in highly complex ores that are widely scattered about the face of the earth, though the demands of recent years have encouraged prospectors to seek promising ore deposits. The chief beryllium ore is beryl ($3BeO.Al_2O_3.6SiO_2$); offering one great handicap at the outset inasmuch as beryllium is present to the extent of only 4 to 5 per cent in the

ore. Other ores exist which have a higher percentage of beryllium, but which in themselves are much scarcer than beryl. Ores of this type are phenacite ($2BeO.SiO_2$) and chrysoberyl ($Be(AlO_2)_2$).

Beryl ore is identified by large hexagonal crystals of a light gray, green, blue, rose, or yellow color. In this wide assortment of colors, and in the sparsity of this ore, absolute identification of beryl is difficult for any but an experienced prospector. A recent authority on the subject of the abundance of metals in the earth's crust[4], lists the occurrence of beryllium as lying between tin and arsenic in abundance. In other words, the available beryllium approaches tin in quantity on the earth's crust, but as will be seen later, basic metallurgical difficulties and the lack of concentrated ore deposits make it very unlikely that beryllium will ever approach tin in commercial quantity.

Among the better known regions of concentrated beryllium ore deposits are the pegmatite dykes of the Winnipeg River area[5] in Canada, over 500 sq. mi., and discovered by field engineers of the Beryllium Development Corp. The largest deposits in the United States lie in the black hills of South Dakota[6], and it it believed that they offer an adequate supply of ore for years to come. To insure an adequate domestic supply of beryl, the export of beryl to foreign countries was prohibited in 1932[7]. Large deposits of beryl have been reported in Eritrea[8]. They are outstanding insofar as a higher percentage of beryllium than in the usual ores is reported. The ore from this last mentioned locality is an amorphous mixture of the metasilicate of beryllium and the more common beryl.

M. J. Moraes dwells at length up the large beryl deposits in Brazil[9]. It is recovered as a by-product from mica, asbestos, and tourmaline mines. The most productive Brazilian beryl deposits are found in the contiguous regions of southern Bahia and eastern Minias Geraes. The largest single crystal of beryl ever discovered was recently found in Brazil. It was 17 ft. by 40 in. and weighed 3 tons. Numerous small flaws throughout the structure nullified the gem value. Though the ores may be scattered about the face of the earth, the supply is adequate for present demands. As will be seen later, the chief concern of beryllium production is centered upon the metallurgical handicaps under which it labors.

Chemical Analysis of Beryllium

The analytical and quantitative determination of beryllium in a mixture of several salts, not readily accomplished by an inexperienced laboratory technician, requires a series of procedures, culminating in the final determination. Commencing with beryl, the usual procedure is to fuse with sodium carbonate, effect a separation of the silicate radical, and precipitate the hydroxides with ammonia. Beryllium is one of the most strongly hydrated, vibalent cations in solution, rendering it very difficult to electro-deposit beryllium from aqueous beryllium salt solutions[10]. This eliminates the possibility of isolating beryllium from aqueous solutions by electrolysis. However, commencing with the hydroxide precipitates above, which contain a mixture of iron, beryllium, aluminum and sometimes manganese, it remains to separate and identify beryllium.

Methods of the separation and the determination of beryllium are outlined carefully in a paper by F. G. Hills[11], in which the methods of Parsons, H. Cooper (in a communication to the referenced author), Lundell and Knowles[12], and his own are compared. Lundell and Knowles perform the comparatively rapid separation of beryllium from aluminum, a step which greatly retarded earlier investigators, by the precipitation of aluminum by 8-hydroxyquinoline from a solution slightly acid with acetic acid. Ferric iron also accompanies a large bulk of the precipitate, leaving beryllium in solution. From this the quantitative determination of beryllium easily follows. As little as 0.05 mg. of beryllium can be detected in a liter by curcumin. A reddish tinge is imparted to the beryllium hydroxide precipitate, but the color fades rapidly.

German investigators for the chemical determination of beryllium are given by Mr. Fischer[13]. Considerable success in rapid determination has been made possible with a great deal of accuracy, by colorimetric titration with quinalizarin (1, 2, 3, 5, 8 tetraoxyanthraquinon). For the quantitative determinations, iron is removed as a complex tartrate, and copper, nickel, and zinc are precipitated from solution as complex cyanides, leaving beryllium in solution.

Beryllium Production Difficulties

An outline of the essential difficulties that accompany the production of beryllium is of value in understanding the methods and precautions that are now in vogue.

1. Beryllium has a tendency to form stable, complex, double salts. The complex beryllium ores emphasize this fact, and the combinations with interfering elements leaves only very little percentage of beryllium. However, there are methods of enriching beryllium ores mechanically. A flotation medium of sodium oleate, pure oil, and lead or barium nitrate has been satisfactory for increasing the ore concentration.[14]

2. There are no known thermic methods that will reduce compact beryllium from its salts. Reductions by carbon have been tried, but the results have been unsatisfactory, hard and brittle beryllium carbide forming.[15]

3. The high melting point of beryllium, in the neighborhood of 1285 deg. C., requires metallurgical processes at high elevated temperatures. It has been pointed out[19] that electrolytic deposits of beryllium from aqueous solutions are extremely difficult. In addition, beryllium sublimes in atmospheric pressure at a temperature sightly higher than 1500 deg. C.[16] Under pressure, the unwarranted loss of beryllium at elevated temperatures may be guarded against.

4. During processes at elevated temperatures, there is the ever present danger of the volatilization of reagents. This necessitates the selection of reagents that will not be lost during the process, due to heat.

5. The fused salts of beryllium are non-conductors of electrical energy, and require the presence of a conducting flux, with which it forms a double salt. Erroneous results were recorded in early researches whereby patent claims were filed for the production of beryllium by the electrolysis of fused beryllium halogen salts—an impossible occurrence unless impurities were present.

6. Beryllium's high affinity for oxygen at elevated temperatures has been noted by all who have undertaken the production of the metal. This is one of the greatest difficulties that has stood in the way of the easy isolation of the metal. Though this fact renders the production difficult at high temperatures, at lower temperatures a beryllium oxide film covers the surface of the metal, rendering it reasonably impervious to corrosive influences.

7. The ultimate yield of the metal is small in comparison to the time and the effort put into its production. This naturally raises the cost of the metal to a great extent.

Commercial Production Methods

The development of commercial methods for the production of beryllium have been concentrated in Germany, United States, and Great Britain. The German methods have been discussed at length in the scientific world[13, 15, 17]; while comparatively little has been written about the details of the American process because of patent litigation. To outline the history of early commercial developments, attention may be called to the personal researches of Stock and Goldschmidt of Germany in 1919, which lead to high temperature electrolysis methods of producing beryllium. A patent for a similar process was issued to Dickinson in the United States, and it is believed in some quarters that this holds precedent over the Stock and Goldschmidt German patent.

The first commercial results are attributed to the Kemet Laboratories of the United States in 1924[18], when ingots of beryllium, produced at great expense, were exhibited at a meeting of the American Electrochemical Society. In Germany, Stock and Goldschmidt attracted wide attention, and the "Beryllium Studiengesellschaft" was organized in 1923 under the leadership of Siemens-Halske, A. G., to conduct an intensive investigation of the production of beryllium and the possibilities of its use. The work was accomplished by 1926, at which time Siemens-Halske, A. G., assumed full control.

In Great Britain, the research on the production of beryllium was headed by Vivian[19], who centered his efforts upon the procurement of beryllium free from impurities. H. Sloman continued where Vivian left off, and attained noteworthy results. In the United States, Prof. Dickinson has already been mentioned as one of the forerunners of commercial production. Another, whose untiring efforts in the field of beryllium have been a source of inspiration is H. S. Cooper, who began his work as early as 1916[20]. His efforts have been absorbed by the Beryllium Corp. of America, now the Beryllium Development Corp., which has made possible large quantities of beryllium for commercial use[21]. At the present day the high cost of commercially pure beryllium stands in the way of wide-spread application of the metal, though it is recognized as a remarkable alloying medium.

One cannot dwell for long upon the production of beryllium without describing the methods of Stock and Goldschmidt, and the improvements rendered by the organized research of Siemens-Halske, A. G. The organization outlined the problems that confronted them[13] as:

1. The high temperature electrolysis method of producing beryllium was deemed feasible.
2. New methods of affording rapid analytical determination of beryllium were necessary.
3. Beryllium ore dressing and the preparation of the salts for the electrolytic process represented the chief item of expense.
4. In 1923, no useful application of beryllium was known.

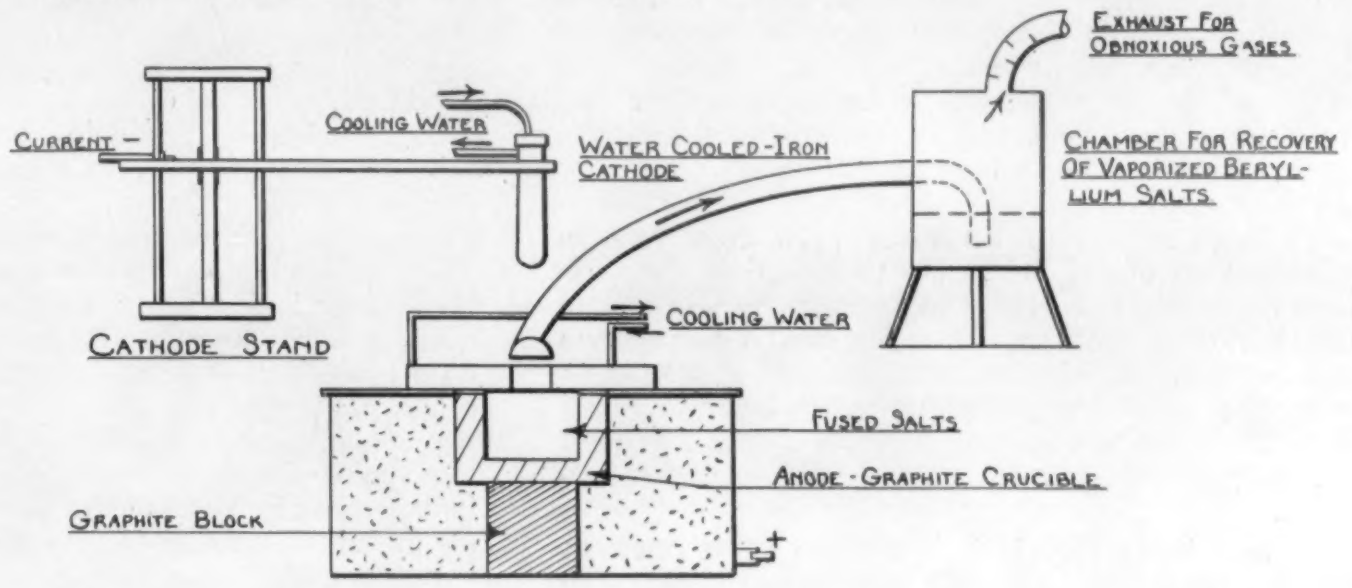


Fig. 1. Production Cell for Metallic Beryllium—German.

PRODUCTION CELL FOR METALLIC BERYLLIUM - GERMAN

5. Beryllium is an unusual metal, with a high melting point and low density (1.84).
6. The effect of beryllium upon heavy metals afforded interesting research.
7. New beryl supplies were necessary to reduce the exorbitant prices.

The original Stock and Goldschmidt patent (DRP.-375, 824-Germany) laid the foundation for the German production[17]. The electrolysis was carried out at a temperature of 1400 deg. C. in a graphite crucible, and with a water cooled iron cathode. A potential of 80 volts was sufficient to keep the salts molten and the temperature of the bath up to its required level. It was not practical to use pure chemical reagents and pure chemical methods due to the great affinity of beryllium for oxygen and carbon at high temperatures, with the subsequent easy formation of impurities. The beryl is sintered with sodium silicofluoride and beryllium sodium fluoride is produced, which is dissolved in water. On treating with caustic lime, a precipitate of calcium fluoride and beryllium hydroxide is produced. Beryllium becomes a water soluble fluoride on treating with hydrogen fluoride, which on evaporating leaves a residue of $5BeF_2.2BeO$. This salt, beryllium oxyfluoride, plays a chief part in the present commercial method of electrolysis in Germany. About two-thirds of the beryllium in beryl is converted into the electrolytic compound[17]. Combined with barium fluoride, it offers low volatility at high temperatures and adequate conductivity for the electrical energy.

No external heat is applied in the German methods of production. A typical production unit is illustrated in Fig. 1[13]. The present process, differing from earlier efforts of Stock and Goldschmidt, heats the crucible by filling with coke, and passing sufficient electrical energy through a compressed graphite electrode to raise the temperature. On reaching dark red heat, at

about 700 to 800 deg. C., the crucible is cleaned by blowing out the coke with compressed air. A small quantity of low melting sodium fluoride and beryllium oxyfluoride is introduced and the electrolysis is started by introducing the cathode. Upon reaching a temperature of 1400 deg. C., above the melting point of beryllium, the cathode is replaced by a fresh one, and equal parts of barium fluoride and beryllium oxyfluoride are added. The electrolysis then proceeds in the normal manner. Beryllium oxy-fluoride is added from time to time, as the process is continuous for 18 to 24 hrs. The anode effect of the fluoride is sufficient to keep the temperature at the required level. During the process the water-cooled iron cathode is slowly withdrawn from the bath (see Fig. 1) and the metal deposited in the form of thick rods, 30 cm. long[17]. The cathodes are replaced frequently as they are covered with beryllium. As early as 1928 large masses of beryllium (150 grams) were produced commercially[15]. Up to that time the German method utilized the double fluoride of beryllium and barium, which was later replaced by beryllium oxy-fluoride, as the barium fluoride accumulates, shortening the time of a single charge. A layer of liquid slag of sodium and barium fluorides covers the fused mixture, affording some protection from extraneous gases.

In explaining Fig. 1, it is well to point out that this represents the highest temperature of commercial electrolytic practice today. The graphite crucible in which the process is conducted is surrounded by another larger one to minimize the large burning loss of graphite, preventing contact between the very hot wall and the open air. The cathode is hollow, permitting the circulation of cooling water. This is necessary in order to solidify the metal into a compact mass (rather than in scales if it were conducted at a lower temperature). The vapor recovery chambers capture insoluble barium-beryllium fluoride and soluble beryllium oxy-fluoride. Obnoxious gases, like carbon fluoride are led to a suitable exhaust remote from the unit. Gases of this nature may seriously endanger the workman's health unless steps are taken to eliminate the hazard. There is a recovery of 90 per cent of beryllium from the fused salts, or about 60 per cent of that present in the original beryl ore[17]. If iron or aluminum are present, they appear as impurities in the beryllium. A modern electrolytic plant for the production of beryllium is now in operation in Germany. The furnaces are covered with hoods to remove the poisonous vapors[22].

Very little has been published about the United States methods of producing beryllium. A patent issued to the Kemet Laboratories in 1927 describes a process whereby beryllium chloride is made conducting by the addition of alkaline earth chlorides. The mixture is melted by the external application of heat. The wrought iron crucible serves as a cathode and a carbon rod as anode. It is important that the melting point of the metal is just reached and not exceeded. Discussion by Merlub and Sobel, following a paper presented by Dr. Illig in 1928 before the American Electrochemical Society, throws a little light on American practice[15]. Beryl is fused with alkali, treated with a fluosilicate, and the beryllium content converted to a salt that is considerably less corrosive than the fluoride used abroad. The electrolysis is carried out at 750 to 800 deg. C. The metal is obtained in the form of flakes or spangels. The electrolysis is carried out at a very low voltage (5 to 6 volts), as compared to the German process. Beryllium oxide is added to lessen the anode effect and lower the electrolysis costs. Sobel felt that the American method was cheaper as there are not as many salts lost in volatilization as in the German process, and at the same time the expenditure of electrical energy is less. However, Dr. Illig points out that the cost of fusing the flakes of beryllium must be added to the production costs of the compact metal. A metal recovery of 75 per cent from the ore to the ingot is claimed[15], and with a purity of 99.25 to 99.5 per cent, as compared with the German 98 per cent purity. It is estimated that the electrolysis by the German method represents one-third of the total cost of the beryllium produced.

The method of producing beryllium in Great Britain was first described in a paper by Vivian[19]. The double fluoride of beryllium and sodium, with an addition of barium fluoride, was used in the electrolysis. The crucible and the electrode were freed of salt impurities by long heating and cleansing methods. Resistance wires brought the crucible to red heat, and on the insertion of two auxiliary anodes into the fused mixture, the application of alternating current brought the temperature of the bath up to 1200 deg. C. At that point the cathode (water-cooled iron rod) is immersed and direct current used. The heat generated on the anode surface by electric arcs maintains the temperature. The cathode is rotated and raised to insure a uniform coat of the metal deposit, at the same time keeping the resistance of the bath to a minimum. During the process, dried nitrogen is introduced into the crucible. Upon completion of the process, the metal upon the cathode is cleaned with caustic soda. For further purification of the beryllium from carbon, the metal is sublimed. The more recent British technique in beryllium production is outlined in some papers by Sloman[42]. These are referred to in the discussion of beryllium alloys.

Table 1.—Comparison on Three Major Methods of Beryllium Production

Characteristic	German (Siemens-Konzern)	United States (Kemet Laboratories)	Great Britain (Vivian)
How obtained	Electrolysis	Electrolysis	Electrolysis
Temperature of electrolysis	1400 deg. C.	750-800 deg. C.	1200 deg. C.
How temperature is arrived at	Elec. res. of coke, then anode effect of reagents	External gas heat	Resistance wire, a.c. thru bath, then anode effect
Cathode	Iron, water-cooled	Graphite crucible	Iron, water-cooled
Anode	Graphite crucible	Carbon rod	Purified graphite crucible
Handling of cathode	Raised	Stationary	Rotated and raised
Essential salt	Beryllium oxy-fluoride	Beryllium (chloride ?)	Double fluoride of beryllium-sodium
Potential of electrolysis	High voltage	Low voltage	High voltage
Estimated yield	60 per cent	75 per cent	.....
Purity of product	98 per cent	99.25 per cent	99.5 per cent
Cost of production (1 = highest)	2	3	1

There is another noteworthy method of producing pure beryllium, but which probably is the most expensive, due to the purity of the salts required[23]. Beryllium salts are dissolved in liquid ammonia and electrolyzed at room temperature. The beryllium salts are dehydrated by fused ammonium salts. The excess ammonium salts are not removed as they lower the vapor tension of the ammonia, and increase the solubility of the beryllium salts. A metal of great purity, resistant to a number of acids, is obtained.

It is interesting to compare the metallurgical

processes for the production of beryllium in England, United States, and Germany. The data are obtained from the literature already given as references, and incorporated in Table 1.

Not only did the British seek to secure especially pure samples of beryllium, but efforts were made to obtain chemically pure beryllium oxide from beryl[24]. Sloman, under the British patent No. 312,007 sought a simple, yet comparatively inexpensive method of extracting beryllium oxide, which would be adaptable to a works scale. He secured beryllium oxide of a high degree of purity (99.4 per cent) from crude by-products, the oxide far exceeding the commercial supply in purity. The beryllium oxide was stored in air-tight bottles because it readily picked up carbon dioxide and water. Beryllium oxide, successfully mixed with cryolite, potassium cyanide, borax, alkaline earths, has been unsatisfactory as a fused electrolyte for beryllium production[17]. Beryllium salts were offered for sale by the Brush Chemical Laboratories in 1932. A price list on beryllium sulfate, beryllium acetate, beryllium nitrate, beryllium chloride, and beryllium oxide was advertised. The Foote Mineral Co., Philadelphia, also advertises the sale of beryllium salts.

Physical Properties

Beryllium, occupying the fourth place in the periodic classification of elements, is one of the lightest metals

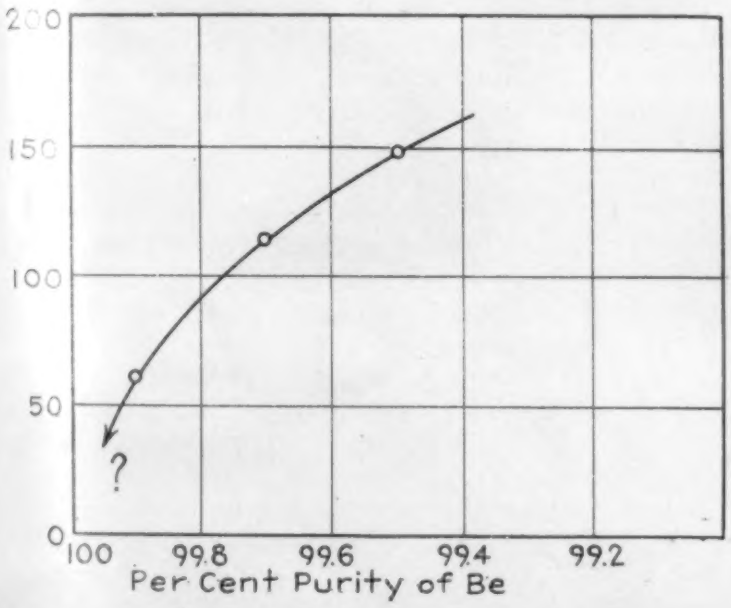


Fig. 2. Relation of Purity to Hardness. (Data—Sloman.)

known. Unusually enough, it has a very high melting point. Numerous investigators are in accord upon the exact value of the melting point of beryllium under atmospheric pressure, placing it at 1280 ± 5 deg. C. The specific gravity is placed at 1.84 (0.065 lbs. per cu. in.)[25]. An approximate comparison of the physical properties of steel, duraluminum, and beryllium is given in Table 2. Of particular interest is the high modulus of elasticity exhibited by beryllium (27,000,000 lbs. per sq. in.), which is so important to structures. Consequently the metal has been suggested for aeronautical construction [26], but the high cost makes this consideration highly improbable. As it is 65 per cent of the weight of duraluminum, beryllium could easily replace it, but the high cost and the extreme difficulty of working anything but the very purest beryllium, renders the use of metallic beryllium improbable.

Table 2.—Approximate Comparison of Physical Properties

Metal	Weight	Strength	Stiffness	Cost	Workability
Steel	100	100	100	Lowest	Good
Duraluminum	35.4	50	33.3	Low	Good
Beryllium	22.7	90	93.0	Very high	Very poor

An important application of pure beryllium is in X-ray tubes. The metal is pressed at red heat into thin plates 20 mm. in diameter and 1 to 2 mm. thick. These plates have a transparency to X-rays, 17 times greater than the current aluminum plates. In addition, Sobel points out that beryllium has been used as small electrodes in neon signals[15].

The great affinity of beryllium for oxygen at elevated temperatures may be represented by the following reaction[17]:

$$\text{Be (solid)} + \frac{1}{2}\,O_2\ \text{(gas)} = \text{BeO (Solid)} + 136{,}000\ \text{calories.}$$

There is a great deal of controversy as to the ductility of beryllium. German and American papers have generally referred to the metal as being "extremely brittle," "harder than quartz," or "easily scratches glass," while the investigations of H. Sloman in Great Britain show a much lower ductility and hardness[27]. As a matter of fact, all of the groups are correct, for the hardness and the ductility are very much dependent upon the degree of impurity of the metal. This fact is well emphasized by referring to Fig. 2. It is apparent that commercially pure beryllium (with a purity of about 99 per cent), would be quite brittle. As Sloman argues, the closely packed, hexagonal crystal lattice of beryllium suggests low ductility on arriving at a sufficiently pure state. Obtaining beryllium of a purity of 99.9 per cent as shown in Fig. 2 is not easy. The beryllium is sublimed in vacuum under 0.1 to 0.5 mm. pressure, at a temperature of 1750 to 1800 deg. C.[27]. The metal is heated by high frequency induction. One of the chief impurities in the metal of this high purity is beryllium oxide.

Thermal Conductivity: The thermal conductivity of beryllium has been established by the following relation[28].

$$\text{Thermal conductivity} = k = 0.3847 + .000751\ t$$
where t is temperature (Centigrade)

Normal Potential: Using beryllium of 99.5 per cent purity, F. Getman established the normal potential of beryllium as —1.13 volts[29]. This was determined from a cell of:

$$\text{Be in } Be(ClO_4)_2\text{—sat'd. KCl — sat'd. } KCl.Hg_2Cl_2 \text{ — Hg.}$$

Linear Expansion: The linear expansion of beryllium (98.9 per cent pure) is less than aluminum, but about the same as steel[13].

The close proximity of beryllium to the lower end of the periodic classification of elements encouraged its use in electronic bombardment investigations[30]. The helium content of beryl has also been investigated in the same light by Walker[31].

In future research it is wise to specify the exact purity of the beryllium that is under investigation, especially when giving the physical properties, inasmuch as the properties are affected profoundly by impurities.

Miscellaneous Applications of Beryllium Oxide

There have been a few efforts to prepare beryllium glass by using beryllium oxide. The first attempt was undertaken by Lai and Silverman in 1932[32]. The

attempt was prompted by the fact that beryllium is in the same group as magnesium, calcium and barium, which are used extensively in glass making. The beryllium oxide is characterized by its low density, transparency, high electrical resistance, and high melting point. Beryllium glass melts at 1400 to 1430 deg. C., higher than other glasses with alkaline earth metals. Beryllium-borate glass (4.4 per cent Be) has been found to be a satisfactory substitute for quartz, insofar as the transmission of ultra-violet light is concerned. In addition, it can be sealed like ordinary glass[33]. A French patent (749,436) utilizes beryl (3-5 per cent) and crystallized quartz for spark plug insulation.

The commercial use of beryllium oxide has been urged, attention being called in particular to its property of retaining insulating characteristics at high temperatures, and its resistance to thermal shock[34]. When made into shapes for refractory purposes, it can be cooled or heated very quickly to 2000 deg. C.

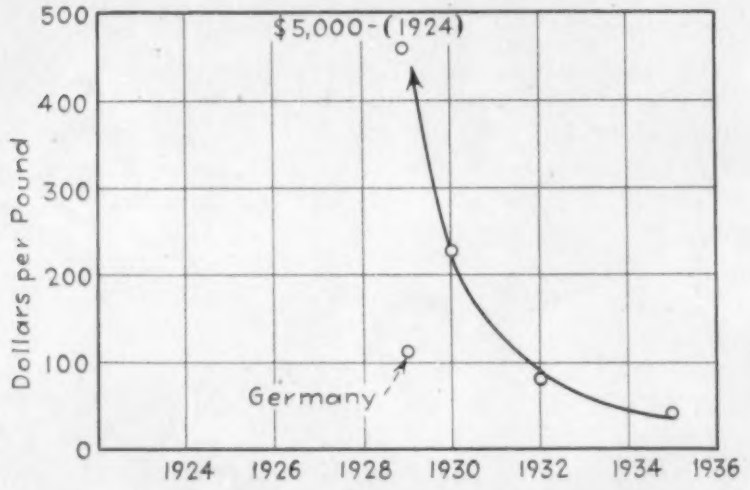


Fig. 3. Market Price of Beryllium in United States.

without cracking. The melting point of beryllium oxide is in the neighborhood of 2570 deg. C.

Price of Beryllium

In the preceding paragraphs there has been no mention of the actual cost of beryllium, as this side of the subject warrants a separate discussion in the light of industrial achievement. The market must be developed to encourage the production of beryllium and lower the price. The use of beryllium as an alloying agent is increasing. During 1933[6], the use of beryllium increased fourfold, and approximately 90 per cent of the domestic consumption was for beryllium-copper alloys. At the end of 1933 the consumption of beryl had reached 35 tons per month. Reference to Fig. 3 will enable one to recognize how improved metallurgical technique and increased consumption have lowered the price of beryllium from $5,000 a lb. in 1924[35] to the present value of $40 a lb. The German price in 1929[36], reported as $108 a lb., is given for comparison with the American price. That occasion was on the eve of the improvements in technique developed by Siemens-Halske, A. G. Since that time, further refinements have lowered the price on both sides of the Atlantic.

Nevertheless, not all the predictions of the future of beryllium are encouraging. Smarting from poor results with beryllium steel research, English editors of technical journals are pessimistic. In an editorial appearing in the "Metallurgist"[37], they urge the discontinuance of the expensive beryllium research on the part of the government, because there has not been a direct yield to British industry as yet. However, that applies only to the British industry, and undoubtedly is due to misguided efforts in attempting to obtain extremely pure beryllium, while the practical production of commercially pure metal has been given secondary consideration. W. Kroll defends beryllium steel research in a letter to the editor[38]. The question is raised again later on discussing beryllium steels. However, the British editors do encourage private industries to finance beryllium research.

As to the future price of beryllium, Dr. Illig estimates that as low as $11 a lb. will be reached[15], while an even more optimistic estimate is made by Dr. Sawyer[39]. A greater demand for the metal, new and cheaper ores, and greater refinements in the metallurgical processes will bring about the reduction in the price of the metal.

(To be continued)

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X-Rays and Atoms

An Industrial Review

By ROBERT C. WOODS

Consulting Engineer, Old Lyme, Conn.

THE DAY IS PAST for guesswork in industrial calculations. Both manufacturer and buyer now ask the same question: "What is it really made of?"

This healthy curiosity has been engendered by discovery that usefulness and durability depend, in the last analysis, not alone on the chemicals in a product, but also on the relations prevailing throughout their innermost units, the atoms. Furthermore, widespread use of new alloys has brought forcibly to the industrialist's attention the fact that elasticity, tensile strength, hardness, etc., are definitely linked with crystal-structure peculiarities.

At the turn of the last century, who could foresee that X-rays would reveal so much of the life and habits of crystals, would solve so many of the puzzles of inter-atomic relations, and would carry us so far within the atom itself? Moreover, there can be no doubt that all three of these advances are of definite industrial value.

It may well be said, in fact, that the study of the various relations existing between matter and X-rays is one of the most important and informative branches of science in the material-analysis field. Industrial applications of X-rays have made possible not only a satisfactory, non-destructive test for subsurface defects, but have opened to us a vaster realm of fact about minute structural details of everyday objects than our ancestors even dreamed existed.

It is proposed here to review certain aspects of X-rays and atoms, and to discuss as simply and non-mathematically as possible some factors relating to this subject.

Although the principle of light-interference has been presented in these pages previously, [February, 1936, pages 45-50], a sentence or two now on the same phenomena will not be amiss. As we know, both radiant energy and matter are in the habit of appearing now as waves and now as particles. For the present let us think of radiation as a system of waves and matter as an aggregation of hard particles.

Proof that light is at times wave-like is based, mainly, on interference experiments which are briefly as follows: A single light-beam, L in Fig. 1, divided at pinholes A and B, will produce alternate light and dark areas on surface X—points like P, equidistant from A and B, will be brightly illuminated by reinforcing, similar-phased waves, but points like Q, unequally distant from A and B, will be unlighted due to the arrival there of cancelling, out-of-phase waves.

Such reinforcements and cancellations due to single-wavelength light travelling different distances can also be caused by reflecting the beam from surfaces at varying distances. Thus, if we have a system of parallel mirrors *a*, *b*, and *c*, as in Fig. 2, which reflect light, of wavelength λ, all waves starting from line AA′A″ and ending at X will not all have covered the same distance.

If we say that the angle at which the light strikes a mirror is θ, and that d is the distance between planes, then route A′B′X is longer than route ABX by twice the distance times the sine of the angle θ (from geometrical considerations). Therefore, although all waves starting from AA′A″ are in phase, they will be in phase on arrival at X only when the distance-difference between them ($2d\sin\theta$) is equal to one exact wavelength, or some whole number of wavelengths—that is, when $2d\sin\theta = \lambda$, or 2λ, or 3λ, or . . . . . $n\lambda$. In other words, when one reflected wave is put out of step with its neighbor, the result is 0.

In 1912, the German physicist, Laue, seeking to prove that X-rays were waves, had the happy thought that atomic spacing within crystals furnished about the proper pinhole-dimensions necessary to show X-ray interference effects. Accordingly, he directed experiments which proved the possibility of diffracting X-rays by crystals, and later, Bragg, using monochromatic rays, devised a method to analyse both the X-ray wavelength—where the crystal spacing was known—

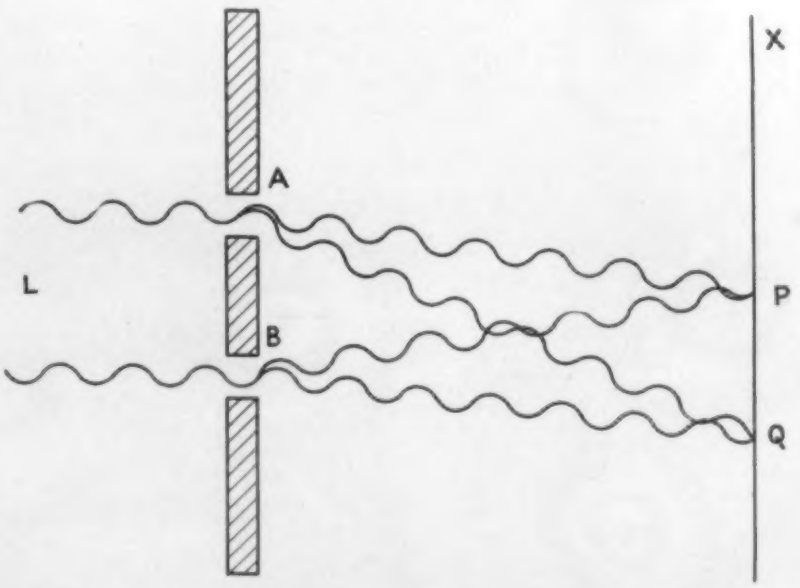


Fig. 1. Light Interference by Diffraction from Pinholes

and crystal structure—where the radiation wavelength was known.

A rough, but graphic, means of illustrating X-ray diffraction by a single crystal is the combination of Figs. 1 and 2 into the crystal-atom picture of Fig. 3, a three-dimensional affair with many planes at different angles. A monochromatic X-ray beam, S, is reflected from the atom-sheets *a*, *b*, and *c*, which compose the

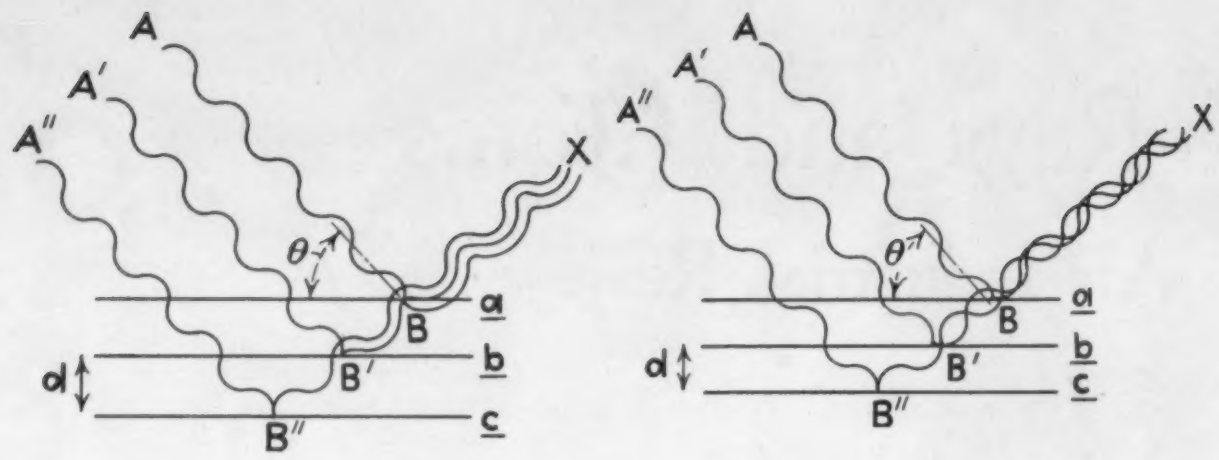


Fig. 2. Light Interference by Reflection from Planes. At left, planes are spaced so that $2d\sin\theta$ equals some whole number of wave lengths. At right, planes are spaced so that $2d\sin\theta$ does not equal some whole number of wave lengths.

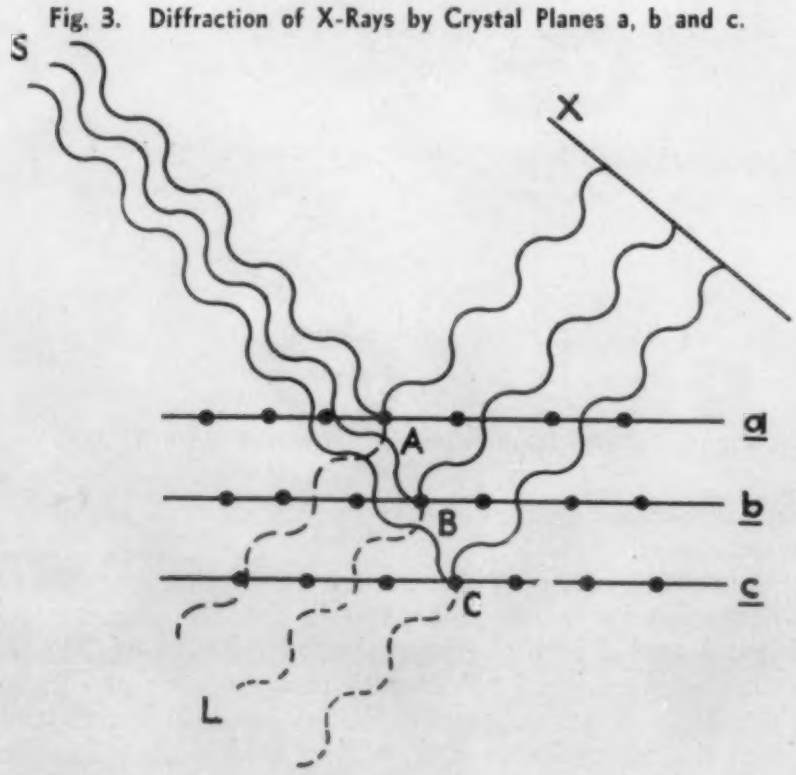
crystal, onto surface X. As in Fig. 2, those sheets so spaced that X-rays are reflected at some whole wavelength-distance ahead or behind other rays, make a diffraction pattern on X characteristic of the crystalline cell.

If it helps any, we may say the result is as though the energy-beam had come from L (see also Fig. 1) and had been diffracted from pinholes at A, B, and C, except that now the pinholes would have to be atomic in size and molecular in spacing to diffract the rays.

Areas of energy-concentration on X are called Laue spots and form a definite pattern around the central, unreflected part of the beam, as is shown in Fig. 4. Position and intensity of the spots indicate size and shape of unit crystal cells as well as the atomic formation. As each element or chemical compound, in general, has its own particular crystal form when solid, we find that the X-ray supplies an accurate way of determining chemical composition, in addition to detecting changes in crystal formation due to external influences. An example of the latter application is the clear demonstration of the crystal differences of body-centered alpha iron and face-centered gamma iron.

At this point the reader may well object that it is not always practical to obtain a single crystal of an industrial material for test. This is easily remedied by use of a powdered sample of the substance, in which case the crystal planes are, of course, pointing in a multitude of different directions, but of this great number of planes some will be fixed properly for X-ray diffraction. Each tiny crystal, so placed, contributes its share of diffracted energy until a cone of rays is formed around the central beam. Further, adjacent atom-layers, chosen at various distances and in different directions, reflect other groups of rays at slightly different angles to form other cones, as in Fig. 5.

Fig. 3. Diffraction of X-Rays by Crystal Planes a, b and c.



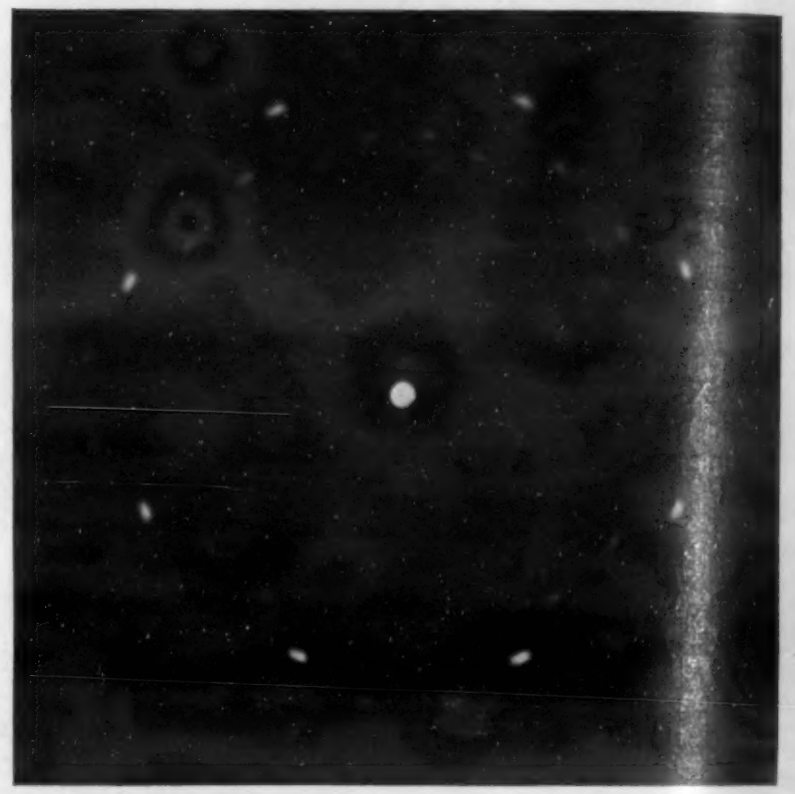


Fig. 4. Laue Pattern Caused by X-Ray Diffraction from Crystal Planes.

The result is a series of concentric interference rings whose positions and intensities, like the Laue spots, give valuable information about the crystal and its atomic members. Filmstrips in Fig. 6 show X-ray powder patterns of copper, sodium chloride, and iron. The central portion of the film is either blocked off or cut out before the exposure is made to avoid the fogging effects of the main beam.

Still another diffraction method yields different, yet important, facts about industrial materials. A diffraction picture of thin samples of materials in the whole state does not reveal so much information about the structure of single crystals, but it does show how the crystals have aligned themselves with respect to one another in the finished product. In this method, a monochromatic beam passed through the sample records reflections from numbers of crystal-faces, as in the powder pattern, except that we have here a smaller number of planes for reflection, and the diffraction is therefore more spotty and irregular. Indication of crystal orientation is of industrial importance, for conditions of strain in materials will be revealed by the X-ray long before the intercrystalline boundaries have slipped sufficiently to be visible under the microscope. In Fig. 7 we have spots caused by

reflection from different crystals in a brass sample, and we can see that the crystals are evidently strewed around at random, the way crystals will combine under natural conditions. Fig. 8, however, shows the crystals, due to rolling, assuming a "spokiness" indicative of

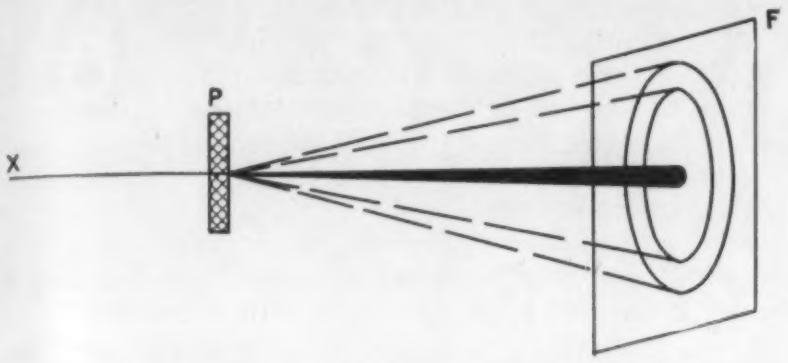


Fig. 5. Interference Rings on Film, F, Caused by Diffraction of X-Ray Beam, X, in Powder Sample, P.

some existing strain which forces the crystals into preferred orientation. Even though this type of X-ray analysis does not give such fundamental facts about crystals, it nevertheless furnishes information of importance in discovery and correction of faulty metal-working processes.

So far, we have taken the use of monochromatic X-rays in crystal-study as a matter of course, but a brief explanation of the production of these single-wavelength rays seems decidedly in order at this point, for it is not only of industrial importance but is also responsible for much of the progress made in atomic physics during recent years.

White light, as everyone knows, contains most all of the wavelengths in the visible spectrum, from about 7000 Angstrom units down to 4000 AUs, and it is only when we split up such light into its component parts that we can distinguish one wavelength from another. In a like manner, beams emitted by ordinary X-ray tubes are really composed of rays spread over a large range, anywhere from 10 AUs to 1/10 AU, and so are usually called beams of "white" radiation. However, light of nearly single wavelength can be obtained from white light by merely interposing, for instance, a red filter in the beam, but no such simple arrangement is satisfactory for separating the various "colors" of a beam of "white" X-radiation. To do the latter, we must use atoms as wavelength-selectors.

For the sake of clearness, it seems wisest to keep closely to mechanistic ideas of atoms in which electrons and nuclei appear as discrete granules of electricity. On this basis, we may, perhaps, picture the simplest of all elements, hydrogen, as in Fig. 9, with N the positively-charged nucleus, and E the negatively-charged electron whirling around N in orbit K. L is another orbit which E might occupy under conditions discussed below.

Now, it is evident that between E and N some force of gravitation or energy of binding must exist—some force which keeps E from flying off at a tangent into space. As this hydrogen atom now stands it is a normal, well-balanced atomic unit and will remain so unless outside influences disturb it. If some blow, however, caused E to move out to orbit L, where it normally has no business to be, the energy-relations between E and N would have to be revised to hold E within the atom. In other words, it requires much more energy to maintain E in orbit L than in orbit K. If, however, such energy is supplied from outside the atom, E will only take up a very temporary position in L and will soon return to its rightful place in K by releasing the extra energy. This type of energy emission is called the characteristic radiation, because the energy absorbed by an electron jumping from its K orbit to the L and then emitted when it jumps back again, assumes a different wavelength for each atom.

Besides the hydrogen K-to-L jumps, there are also other jumps in other atoms—for example, L-to-M, M-to-N, etc.—and each atom has its characteristic wavelength, characteristic of the orbit emitting it. Furthermore, it has been proved that this absorption and emission of energy does not take place in a smooth, continuous way, as we should expect, but occurs in

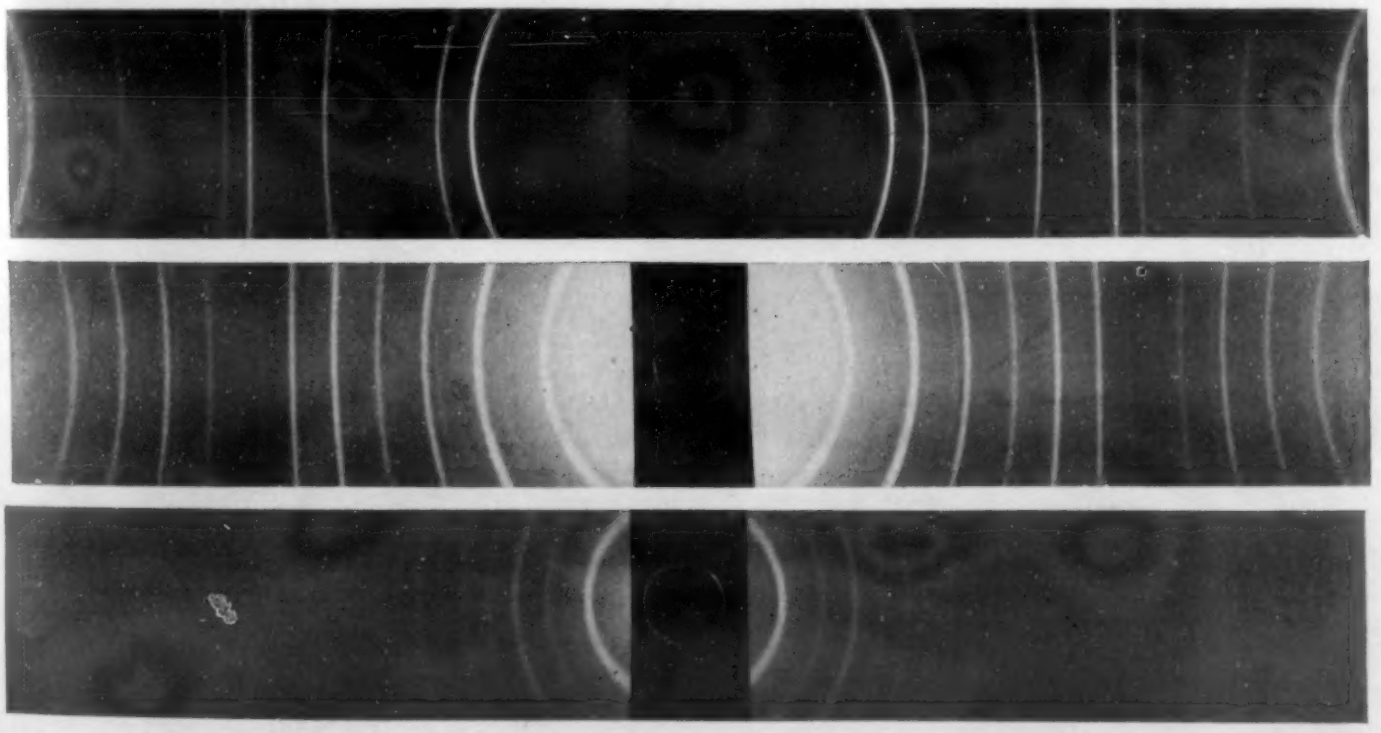
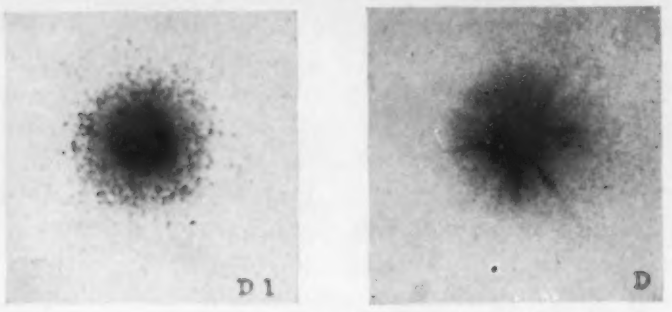


Fig. 6. Interference rings on Film Strips by X-Ray Diffraction in Powder Samples of Copper, Sodium Chloride and Iron.

"fits and starts," as if the energy were done up in little indivisible packets, or bundles. So particle-like is this process, in fact, that each wavelength ray can be accurately predicted to contain an amount of energy obtained by multiplying its frequency, ν times Planck's constant, h, which is equal to 6.547×10^{-27} erg-seconds. It is further found that, in any given atom, E can be moved from K to L only by incoming energy of a certain $h\nu$—all other $h\nu$'s having no effect. It

almost seems as though E were a very fussy person that would not touch food unless it were done up in a certain way. These tiny energy packets are called quanta and upon their existence is based the whole structure of quantum mechanics.

Accordingly, if a given beam of X-rays has a frequency 5000 times as great as, say, green light, then the ν in the $h\nu$-bundle will be 5000 times as large, and each X-ray quantum will contain 5000 times as much energy.



Figs. 7 and 8. Diffraction Patterns of Brass. At left, crystals show good random orientation. At right, crystals show "spokiness" due to strain.

All this may seem, at first glance, very far from the routine process of a steel or brass analysis, but in reality it forms the background of the whole matter. While it is true that hydrogen spectrum quanta, such as we described, are low in energy value or concentration—that is, are in the region of visible light—spectra of heavier atoms bombarded in an X-ray tube by high-speed, high-energy, electrons, give quanta in the high-frequency range of X-rays.

In 1913, the 27-year-old English scientific genius, Moseley, whose brilliant career ended in the trenches in Gallipoli, found that when various atoms, or elements, were used as targets in X-ray tubes, the frequencies of the emitted rays increased in a simple arithmetical progression as the atomic number of the bombarded element rose. While it would be more accurate to say that the square root of the frequencies is thus related to atomic number, the important point is that the atomic properties responsible for this Moseley effect are the internal electronic structure and the linearly increasing charge on the nucleus. This knowledge, then, supplies us with a method of producing monochromatic X-rays of almost any desired wavelength, just as we can produce at will green, blue, or violet light, for we can produce the wavelength ray associated with any atom by merely making a target out of the desired element and bombarding it with electrons of the correct energy. Thus when a copper target is bombarded, the emitted X-rays are about 1.5 AUs, while with molybdenum the wavelength is 0.7 AU.

These X-ray spectra characteristic of the atom emitting them can be recorded on film, and Fig. 10 shows spectra from targets of copper, molybdenum, and tungsten, the notations indicating the atom-orbit responsible for each wave or quantum.

In conclusion, we have seen that X-ray studies supply us with all kinds of facts about formations and details of crystal structure, show us the relations existing between atoms of similar or dissimilar substances, and furnish us with a probe to explore deep within the atom itself.

Such a discussion as this can, of necessity, cover only the more elementary aspects of the subject and cannot be expected to include those abstruse, mathematical theories with which modern physics abound. Like our ancestors of the Stone Age, we still want to draw

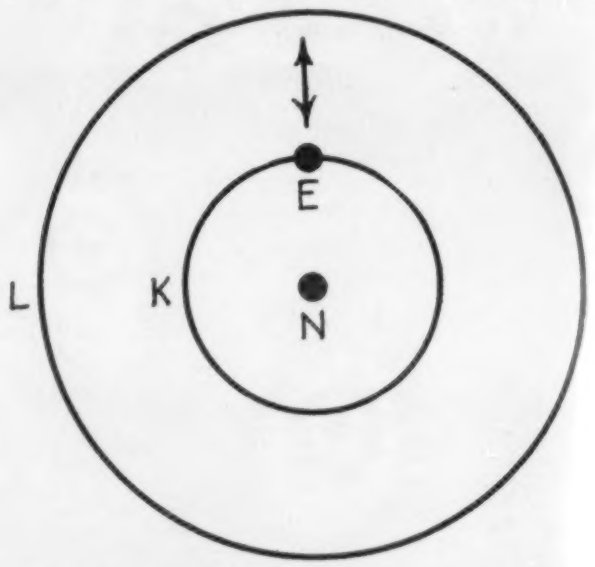


Fig. 9. Graphic Representation of Hydrogen Atom. Electron E revolves in orbit K around nucleus N. L is orbit E might occupy when excited.

diagrams and paint pictures of our ideas of Nature, even though it seems likely that Nature itself is quite indifferent to the success of such attempts. Nevertheless, we are entitled to view our models and drawings as stopping-off places where we rest for a moment before going on. Einstein recently said, "In spite of the fact that today we know positively that classical mechanics fails as a foundation dominating all physics, it still occupies the center of all our thinking in physics.

"The reason for this," he continues, "lies in the fact that we have not yet arrived at a new foundation in physics concerning which we may be certain that the whole complexity of investigated phenomena could be deduced logically from it."

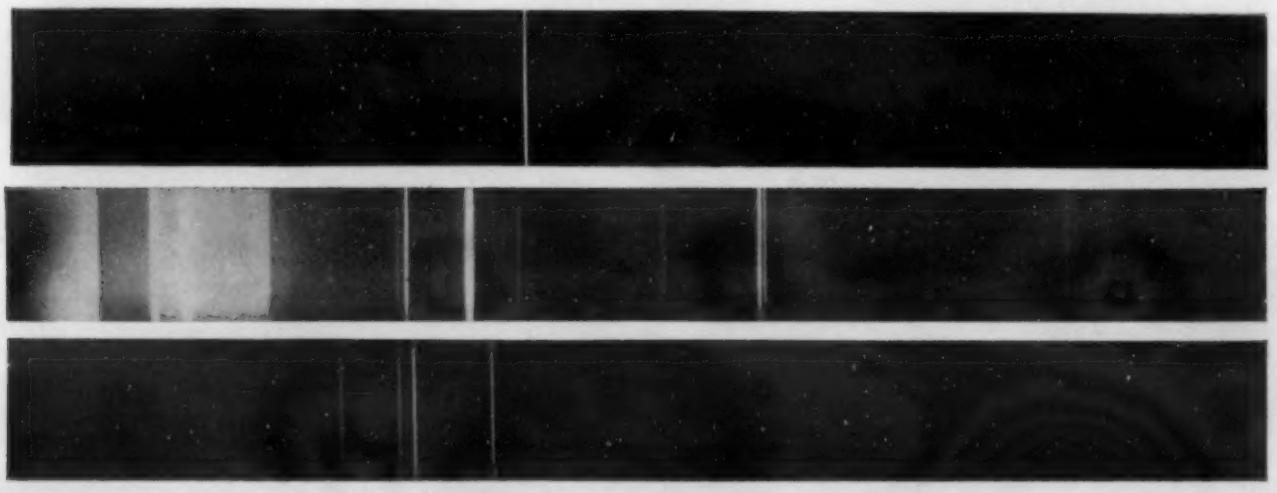


Fig. 10. X-Ray Spectra of Copper, Molybdenum and Tungsten. Linespacing shows characteristic wave lengths (or frequencies).

Nickel Steels at Low Temperatures—II

An Investigation of the Impact Properties of the 2¼ Per Cent Steels

By B. G. ALDRIDGE AND GEORGE C. SHEPHERD, JR.

Development and Research Departments, Respectively, of the Union Oil Co. of California.

Concluded from the June Issue

Problems Involved in Fusion Welding

In the manufacture of the pressure vessels by fusion welding from rolled steel plates of nickel steel, the following questions immediately arose:

What effect would the direction of the mill rolling have on the impact values?
What type of heat treatment should be followed?
What would be the effect of welding on the impact values?
What would be the effect of plate thickness on the impact values?
What would be the effect on the impact values of the steel when held at low temperatures for extended periods of time?

As several investigators had previously reported some differences in the impact values due to the directional properties of rolled plate steel, specimens of a nickel steel plate were taken with their major axis both transverse and longitudinal to the direction of rolling. The effect of the location of the axis of the keyhole notch with respect to the plane of rolling was also investigated. The methods of taking the specimens are illustrated graphically in details A, B, C, and D of Fig. 9. The data in Table 5 shows the effect of specimen location in the plate on the impact properties. It will be noted that impact values approximately 25 per cent higher throughout the entire temperature range tested are obtained from specimens with their major axis longitudinal to the direction of rolling, as compared to those with their major axis transverse to the direction of rolling. It will be noted that the location of the keyhole notch has little, if any, effect on the average impact values obtained.

In the fabrication of the vessels the procedure followed in the heat treatment of the steel plates was to heat the plates up to a temperature just above the thermal critical point for normalization, hold at this temperature for 1 hr., then air cool as rapidly as possible in the open. The plates were then shaped and welded, and on completion of the vessels, they were stress relieved at a temperature of 1200 deg. F.

Table 5.—Effect of Direction of Rolling on the Impact Strength of Nickel Steel Plate

All tests made on standard ASST Charpy impact specimens. The plate was normalized at 1450 deg. F. for 1 hr. and drawn at 1200 deg. F. for 1 hr.

Relation of Major Axis of Specimen to Direction of Rolling	Relation of Axis of Keyhole Notch to Plane of Rolling	Temp. of Test, deg. F.	No. of Tests	Charpy Impact Value, Ft.-lbs., Aver.
Longitudinal	[a]	70	8	43.7
Transverse	[a]	70	8	35.3
Longitudinal	[a]	—50	16	28.5
Transverse	[a]	—50	16	22.8
[b]	Parallel	70	8	39.8
[b]	Perpendicular	70	8	39.2
[b]	Parallel	—50	16	25.8
[b]	Perpendicular	—50	16	25.6
[b]	[a]	70	24	39.5
[b]	[a]	—50	24	25.7

[a] One half the notches were parallel, and the other half were perpendicular.
[b] One half the specimens were taken longitudinally, and the other half were taken transversely.

Effect of Normalization

The effect of normalization at various temperatures on the impact strength of 2.32 per cent nickel and 0.30 per cent carbon 1 1/8-in. steel plate is shown in Table 6, and portrayed graphically in Fig. 10. It will be noted that stress relieving at 1200 deg. F. only does not materially improve the impact values over those obtained on the plate in the as-rolled condition, but that normalization at just above the upper thermal

Table 6.—Effect of Heat Treatment on the Impact Strength of Rolled Nickel Steel Plate

All tests made on standard ASST Charpy impact specimens. The tests were made on specimens cut from sections of 1⅛-in. nickel steel plate. The specimens were all taken transversely to the direction of rolling. The tests were all made at —50 deg. F.

Normalization		Draw		Number of Tests	Charpy Impact Value, Ft.-lbs.		
Temperature	Hrs.	Temperature	Hrs.		Min.	Max.	Aver.
....	..	....	..	4	12.2	15.5	13.9
....	..	1200	1	4	13.4	16.1	14.7
1450	1	1200	1	4	17.4	21.9	19.0
1450	2	1200	1	4	16.3	17.9	17.2
1500	1	1200	1	4	13.2	17.8	14.7
1500	2	1200	1	4	4.7	17.8	14.1
1500	3	1200	1	4	10.5	16.8	13.6
1500	4	1200	1	4	4.5	14.5	10.0
1550	1	1200	1	4	11.4	16.3	13.8
1550	2	1200	1	4	4.0	17.3	12.2
1600	1	1200	1	4	3.2	9.9	6.1
1700	1	1200	1	4	2.6	3.9	3.3

The plate had the following chemical analysis: Carbon 0.30, manganese 0.65, silicon 0.18, nickel 2.32 per cent.

Table 7.—Effect of Heat Treatment on the Charpy Impact Properties of Specimens from Rolled Nickel Steel Plate

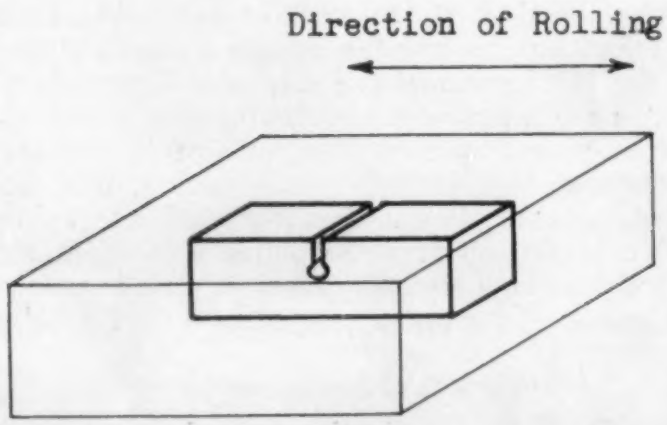
(These tests were made on a 1⅜-in. Lukens plate of the following analysis: Carbon, 0.25%; nickel, 2.25%.)

Specimen Designation	Normalize, deg. F.	Renormalize, deg. F.	Draw, deg. F.	Relationship of Major Axis of Specimen to Direction of Rolling	Charpy Impact Value, Ft.-lbs.					
					70 deg. F.			—50 deg. F.		
					Test 1	Test 2	Aver.	Test 1	Test 2	Aver.
AS	....	....	....	Longitudinal	32.1	34.6	33.4	17.7	20.9	19.3
BS	....	....	....	Transverse	28.3	28.1	28.2	15.2	16.0	15.6
AS-BS	....	....	....	Average	...	...	30.8	...	...	17.4
CS	....	....	1200	Longitudinal	35.2	33.7	34.4	22.9	19.8	21.4
DS	....	....	1200	Transverse	26.7	28.6	27.6	17.7	16.7	17.2
CS-DS	....	....	1200	Average	...	...	31.0	...	...	19.3
ES	1450	....	1200	Longitudinal	39.6	50.5	45.0	30.0	26.0	28.0
FS	1450	....	1200	Transverse	39.6	38.0	38.8	24.6	21.9	23.2
ES-FS	1450	....	1200	Average	...	...	41.9	...	...	25.6
GS	1700	1450	1200	Longitudinal	38.0	41.3	39.6	27.4	28.8	28.1
HS	1700	1450	1200	Transverse	34.0	31.8	32.9	20.8	21.1	21.0
GS-HS	1700	1450	1200	Average	...	...	36.2	...	...	24.6
IS	1700	....	1200	Longitudinal	28.7	...	28.7	6.4	...	6.4
JS	1700	....	1200	Transverse	24.0	...	24.0	3.6	...	3.6
IS-JS	1700	....	1200	Average	...	...	26.4	...	...	5.0

critical point results in a marked improvement in impact properties. On heating to temperatures above this, a degradation in impact value occurs, and at temperatures above 1550 deg. F., the impact value falls off rapidly to very low figures.

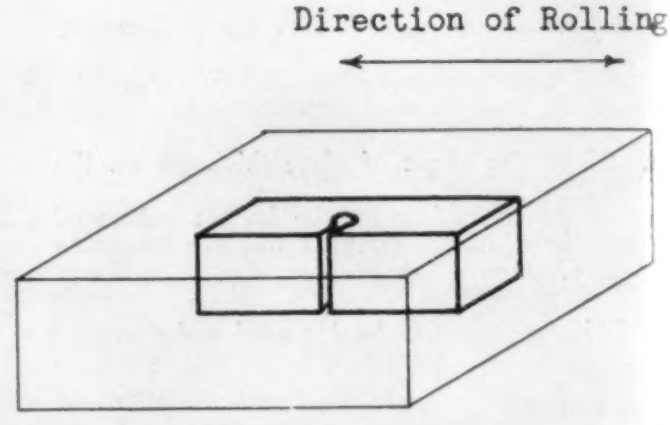
The results throughout on this plate are rather low, and it is believed that this is due to its relatively high carbon content. It will also be noted that the impact values fall off as the specimens are held for longer periods of time at the normalization temperature. Views A, B, C, and D of Fig. 11 shows the effects or grain size on the impact value. It will be noted that the more refined grains give the higher impact value, and as the grain size is increased, either by prolonged heating or by higher temperatures, the impact value falls off quite rapidly.

Fig. 9. Sketches Showing Various Methods of Obtaining Impact Specimens from Rolled Plate.



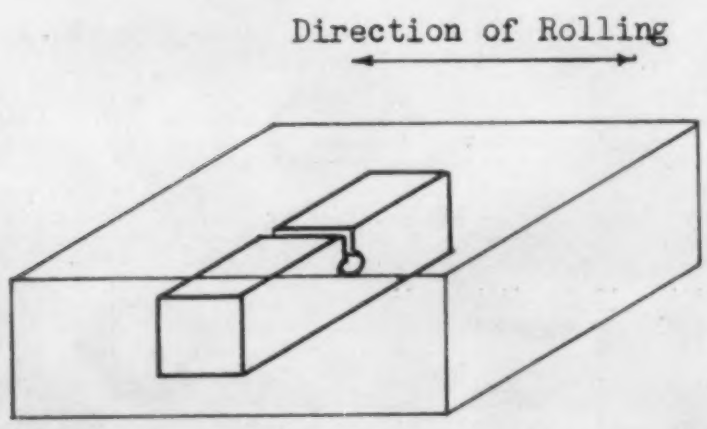
Detail A

Major axis of specimen longitudinal to direction of rolling. Axis of keyhole notch parallel to plane of rolling.



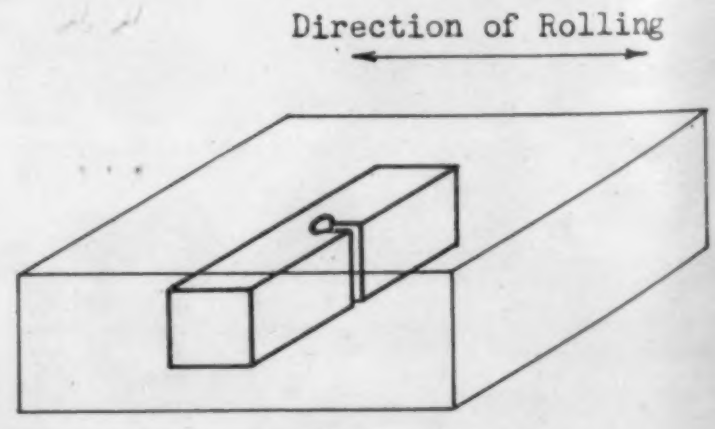
Detail B

Major axis of specimen longitudinal to direction of rolling. Axis of keyhole notch perpendicular to plane of rolling.



Detail C

Major axis of specimen transverse to direction of rolling. Axis of keyhole notch parallel to plane of rolling.



Detail D

Major axis of specimen transverse to direction of rolling. Axis of keyhole notch perpendicular to plane of rolling.

Supplementary tests were made to determine the effect of heat treatment on a 1 3/8-in. steel plate of 2.25 per cent nickel and 0.25 per cent carbon content. The results of these tests are shown in Table 7. It will be noted that stress relieving at 1200 deg. F. only very slightly improves the impact values over those obtained on the plate in the "as-rolled" condition, but that normalization at just above the upper thermal critical point results in a marked improvement in impact properties over the entire temperature rang. Apparently the directional impact properties are rather persistent and not easily destroyed by heat treat-

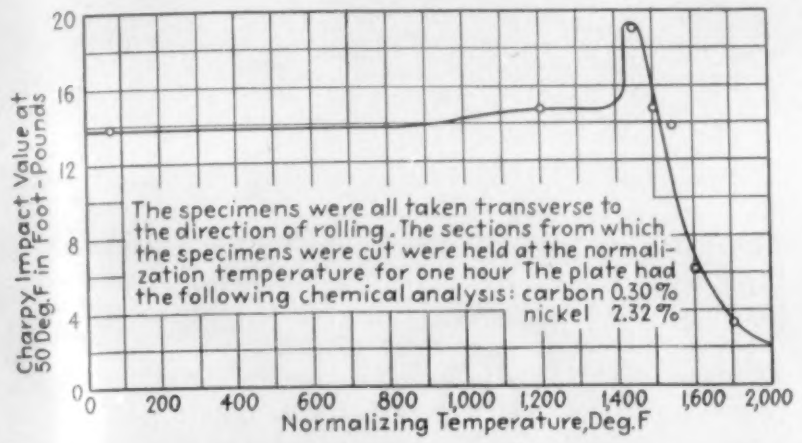


Fig. 10. Effect of Heat Treatment on the Impact Strength of Rolled Nickel Steel Plate.

ment, for even after heating to 1700 deg. F., and then renormalizing at 1450 deg. F., the directional impact properties are still quite marked. The tests on the plate that had been normalized at 1700 deg. F. and then renormalized at 1450 deg. F. show the possibility of restoring the impact value even after it has been destroyed by improper heat treatment or overheating.

Table 8.—Impact Strength of Metal Taken from Various Locations In a Welded Nickel Steel Plate

All tests made on standard ASST Charpy impact specimens. The specimens were taken from various locations from a 1⅜-in. nickel steel plate, normalized at 1500 deg. F., scarfed, welded, and stress relieved at 1200 deg. F. The axis of the welding groove was transverse to the direction of rolling.

Specimen Location [a]	Section of Welded Plate	Relationship of Major Axis of Specimen to Direction of Rolling	Relation of Axis of Key-hole Notch to Plane of Rolling	Charpy Impact Value, Ft.-lbs. 70 deg. F.	Charpy Impact Value, Ft.-lbs. —50 deg. F.
A	Plate	Longitudinal	Parallel	35.0	21.8
B	Plate	Longitudinal	Perpendicular	34.8	22.4
C	Plate	Transverse	Parallel	30.2	17.9
D	Plate	Transverse	Perpendicular	29.7	18.3
E	Affected Zone	Longitudinal	Parallel	51.7	36.1
F	Affected Zone	Longitudinal	Perpendicular	47.1	36.0
G	Affected Zone	Transverse	Parallel	39.6	20.4
H	Affected Zone	Transverse	Perpendicular	35.2	20.1
I	Fusion Line	Longitudinal	Parallel	32.0	26.6
J	Fusion Line	Longitudinal	Perpendicular	31.2	21.9
K	Fusion Line	Transverse	Parallel	31.8	20.1
L	Fusion Line	Transverse	Perpendicular	32.0	21.6
M	Weld	Longitudinal	Parallel	33.7	21.9
N	Weld	Longitudinal	Perpendicular	34.8	23.6
O	Weld	Transverse	Parallel	33.2	22.4
P	Weld	Transverse	Perpendicular	34.5	23.2

[a] See Fig. 12.

In the fabrication of the vessels by fusion welding, tests were made to determine the impact value of the welded plates in the weld and adjacent to the weld. Test plates were taken from plates 1⅜-in. thick, and were normalized at 1500 deg. F., scarfed, welded, and stress relieved at 1200 deg. F. The plates were welded in such a position that the seam was transverse to the direction of rolling, and the welding technic, as closely as possible, duplicated the conditions encountered in commercial welding. The plates were held rigid, magnetic conditions were carefully controlled (DC welding was used), the welding groove

Table 9.—Impact Strength of Nickel Steel Weld Metal

All tests made on standard ASST Charpy impact specimens from the weld metal obtained with nickel steel weld rod when welding two nickel steel plates. The welded joint was stress relieved at 1200 deg. F. The plates were welded so that the welding groove was transverse to the direction of rolling.

Relationship of Major Axis of Specimen to Major Axis of Welding Groove	Relationship of Axis of Keyhole Notch to Surface of Weld	Test Temp., deg. F.	No. of Tests	Charpy Impact Value, Ft.-lbs. Min.	Max.	Aver.	Aver. Deviation
Longitudinal	Parallel	70	3	29.4	36.3	33.2	2.5
Longitudinal	Perpendicular	70	3	33.7	35.1	34.5	0.5
Transverse	Parallel	70	3	30.2	37.3	33.7	2.3
Transverse	Perpendicular	70	3	33.4	36.1	34.8	1.0
Longitudinal	Parallel	0	3	23.2	31.6	27.5	2.9
Longitudinal	Perpendicular	0	3	26.9	29.4	28.2	0.9
Transverse	Parallel	0	3	23.5	30.0	26.9	2.3
Transverse	Perpendicular	0	3	28.1	29.5	28.8	0.5
Longitudinal	Parallel	—75	3	19.4	26.6	22.4	2.8
Longitudinal	Perpendicular	—75	3	21.8	24.2	23.2	0.8
Transverse	Parallel	—75	3	18.7	25.5	21.9	2.4
Transverse	Perpendicular	—75	3	22.9	24.2	23.6	0.5

was standard, and the time interval of bead disposition was similar to plant practice. A coated welding rod of approximately the same composition as the plate was used. Fig. 12 shows the location of the various Charpy impact specimens which were taken from the plates.

The results of these tests are summarized in Table 8. Each value represents the average of at least two determinations. It is well to note here that the plate was normalized at 1500 deg. F., and due to later developments, it is believed that if the plate had been normalized at 1450 deg. F., the impact results on the plate material remote from the weld would have been approximately 5 ft.-lbs. higher.

The affected zone is that portion of the base plate adjacent to the fusion zone whose microstructure has been modified by the heat (probably 3000 deg. F.) generated in welding. The portion of the plate where the temperature is raised above the thermal critical point of the steel recrystallizes. The heat conductivity of the plate is such that this is, in effect, a quenching operation and tends to produce grain refinement. The photomacrograph shown in view A of Fig. 13 shows the base plate, the fusion zone, and the weld. It will be noted that the affected zone is relatively narrow, ordinarily between 1/8 and 3/16 in. wide.

View B of Fig. 13 shows the normal plate microstructure and view C of Fig. 13 shows the affected zone microstructure. It should be noted that considerable grain refinement has taken place. The specimens taken longitudinal to the direction of rolling

Table 10.—Summary of Results of Charpy Impact Tests on Specimens from Sections Removed from Vessel Openings of Union Oil Company Nickel Steel Pressure Vessels

(Major axis of all specimens taken transversely to the direction of rolling.)

Specimen Location	Temperature of Test, deg. F.	Number of Tests	Charpy Impact Value, Ft.-lbs. Min.	Max.	Aver.
Shell	0	35	24.2	38.0	31.3
	—50	35	20.8	34.9	26.5
	—75	35	17.2	31.5	24.4
Head	0	17	24.0	45.2	31.4
	—50	17	20.4	33.2	26.0
	—75	17	16.5	27.6	22.7
Neck	0	10	23.5	38.0	31.8
	—50	10	20.8	30.3	26.4
	—75	10	21.6	29.8	24.0
Cone	0	1	24.6	24.6	24.6
	—50	1	22.9	22.9	22.9
	—75	1	17.9	17.9	17.9
All	0	63	23.5	45.2	31.4
	—50	63	20.4	34.9	26.4
	—75	63	16.5	31.5	23.8

(transverse to the welding groove) have appreciably higher impact values than the transverse specimens. This is due not only to the direction properties of the plates, but also is due to the fact that the notch of the longitudinal specimen lies entirely in the affected zone, whereas the notch of the transverse specimen straddles it.

Tests were made to determine if the change from weld metal to the plate at the fusion line had any effect on the impact strength. In general, quite satisfactory results were obtained, but due to the extreme narrowness of the zone, there is probably some overlap into the weld metal and affected zone.

Table 9 shows the impact strength of the weld metal obtained in welding the nickel steel plates. It will be noted that, in general, satisfactory impact values are obtained, and the relation of the major axis of the welding groove is of little significance. The specimens with the axis of the keyhole notch perpendicular to the plane of bead deposition show somewhat higher impact values than do the specimens in which the axis of the keyhole notch is parallel to the plane of bead deposition. This is probably due to a tendency to unidirectional crystal orientation in the cast "as welded" structure, which is not entirely modified by the heat-treating effect of subsequently deposited beads and also a variable heat-treating effect from the top of the bead through to the bottom of the bead.

Also it will be noted that much more consistent results are obtained with the perpendicular notch than with the parallel notch. The parallel notch specimens are representative of 5 mm. of weld metal depth,

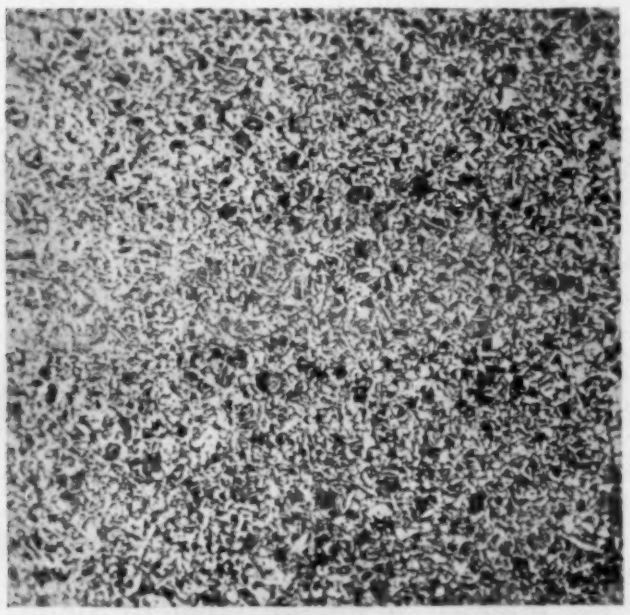
Table 11.—Effect of Heat Treatment on the Charpy Impact Properties of Cast Nickel Steel

(These impact values were obtained by the Crane Co. laboratories on cast nickel steel of the following chemical composition: Carbon, 0.24%; nickel, 2.00%.)

Heat Treatment Temperatures			Temp. of	Charpy Impact Value, Ft.-lbs.		
Normalization, deg. F.	Renormalization, deg. F.	Draw, deg. F.	Test, deg. F.	Test 1	Test 2	Aver.
....	....	....	70	22.0	22.5	22.2
....	....	....	0	11.0	...	11.0
....	....	....	—50	8.5	8.5	8.5
....	....	....	—75	3.5	5.0	4.2
1450	....	1200	70	28.0	33.0	30.5
1450	....	1200	0	21.0	21.5	21.2
1450	....	1200	—50	21.0	20.5	20.8
1450	....	1200	—75	7.0	14.5	10.8
1725	....	1200	70	32.0	35.0	33.5
1725	....	1200	0	27.0	27.0	27.0
1725	....	1200	—50	22.0	23.0	22.5
1725	....	1200	—75	20.5	21.0	20.8
1725	1510	1200	70	37.5	39.0	38.2
1725	1510	1200	0	27.0	29.5	28.2
1725	1510	1200	—50	22.5	25.5	24.0
1725	1510	1200	—75	23.0	...	23.0

whereas the perpendicular notch specimens will be representative of 10 mm. of weld metal depth. The perpendicular specimens should therefore be more representative of the weld metal. View D of Fig. 13 shows the transition from the finer-grained to the coarser-grained weld metal in the upper part of the weld. The columnar structure of the weld metal is also evident in this photomicrograph. Stress relieving the weld metal was found to improve its impact strength approximately 20 per cent at —50 deg. F.

Table 10 summarizes the results of the Charpy impact tests on specimens taken from the metal cut from vessel openings of over 4 in. in diameter after



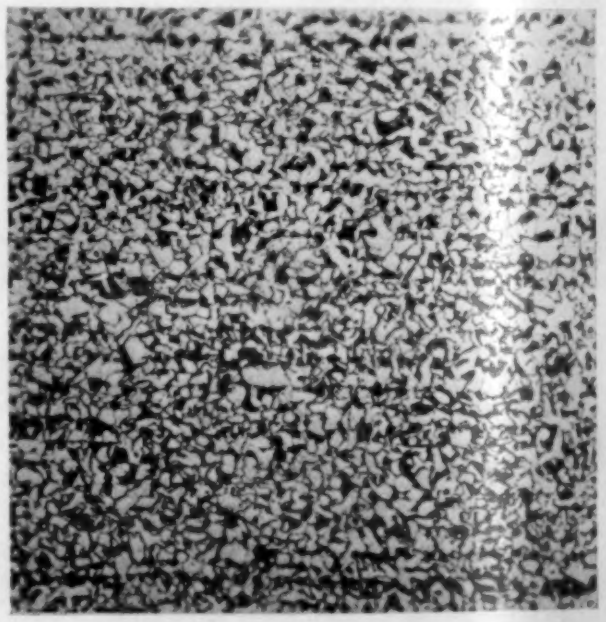
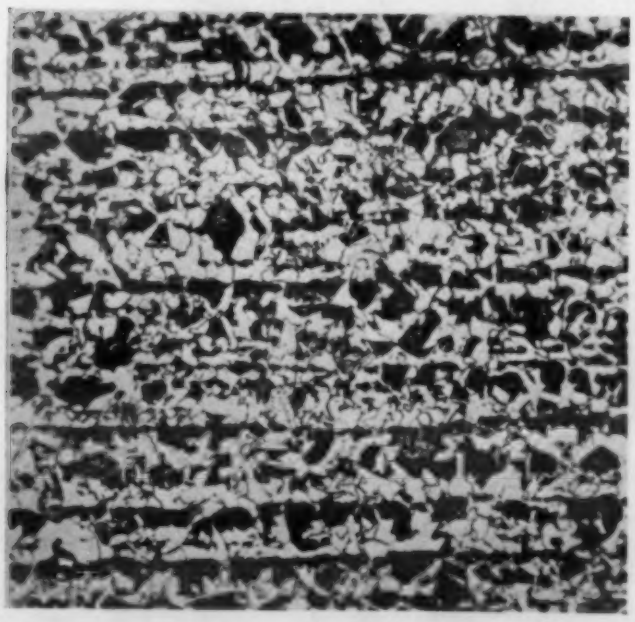
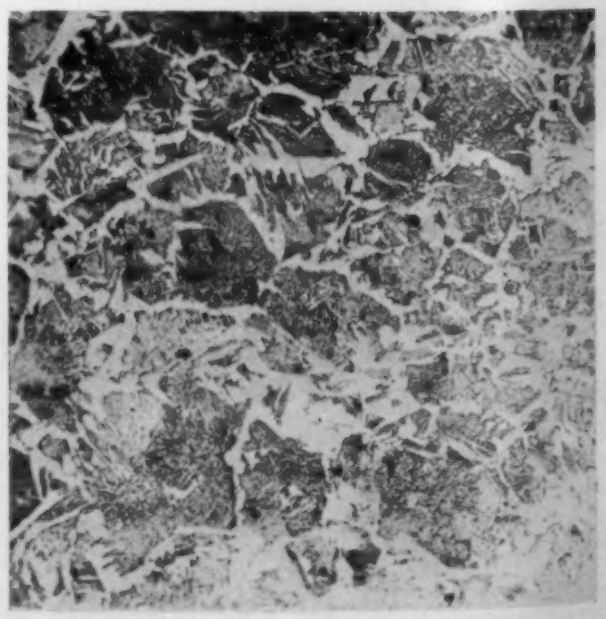


Fig. 11. Effect of Grain Size on Impact Value.

View A—(Left) Nickel Steel Plate 31.8 ft. lb. at -50 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

View B—(Right) Nickel Steel Plate 23.7 ft. lb. at -50 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.





View C—(Left) Nickel Steel Plate 15.4 ft. lb. at -50 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

View D—(Right) Nickel Steel Plate 3.3 ft. lb. at -50 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

the vessels had been fabricated, and also summarizes the result of the impact tests in the weld metal from the test plates, two of which were welded integrally with the longitudinal welds of one of the ring sections of each of the 16 nickel steel vessels.

To determine what, if any, was the effect of plate thickness upon the impact values of steel, plate tests were made on plates ranging from 5/8-in. thickness up to 1 3/8-in. thickness. The test specimens were taken from near the surface and center of the plates, but no significant differences could be detected due to the location of the specimens, and over the range of plate thicknesses tested, the results indicated that the thickness of the plates had no effect upon the impact value. This, however, might possibly not be the case were the plates considerably thicker than those tested. Specimens taken from the top, bottom, sides and center of these plates showed little variation.

As certain investigators had reported a marked diminution in impact values after the specimens were held for 12 or more hours at low test temperatures, additional tests were made to determine this effect. These tests were made by maintaining the specimens in a bath at —50 deg. F. for various periods of time, ranging from 20 mins. to 72 hrs. Little, if any, difference was discernible in the average impact values obtained on the specimens.

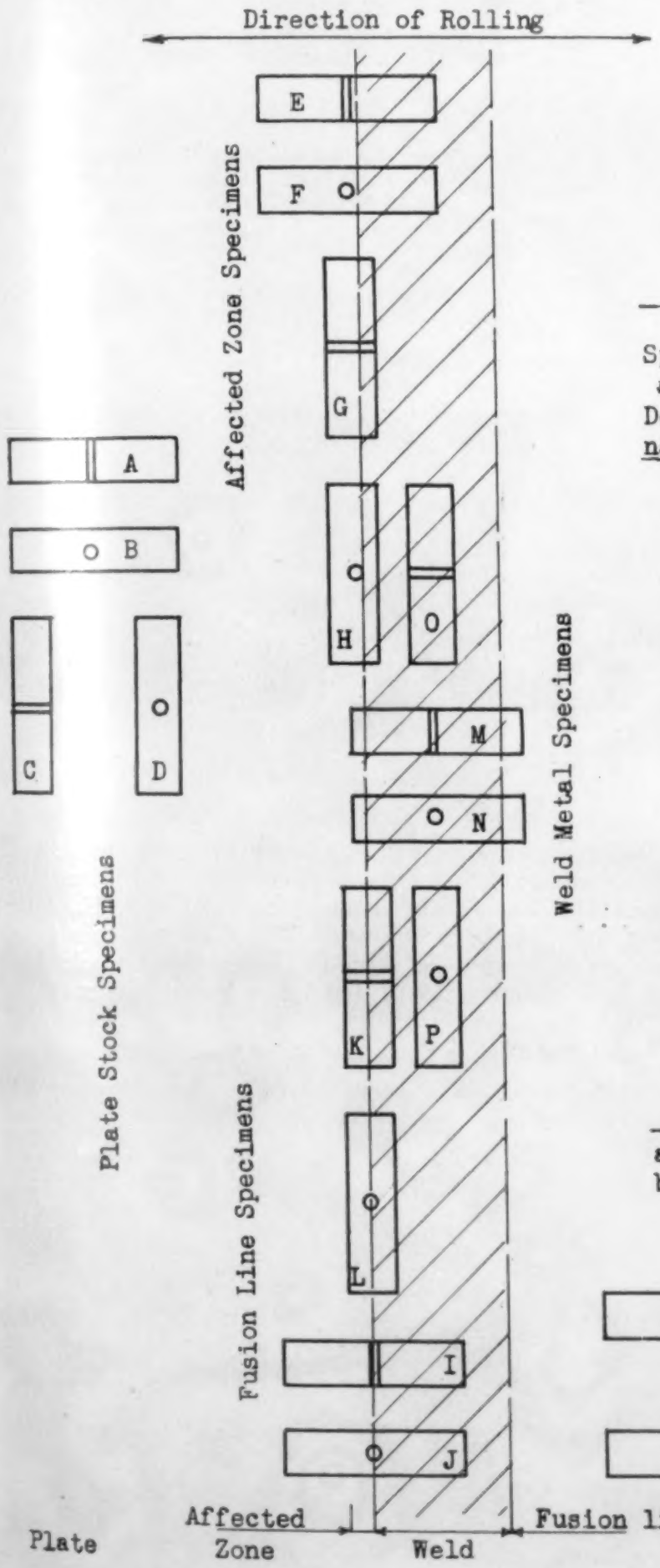


Fig. 12. Diagrammatic Sketch showing How Impact Specimens Were Taken.

Specimen Designation	Location of Specimen	Relation of Major Axis of Specimen to Direction of Rolling	Relation of Axis of Keyhole Notch to Plane of Rolling
A	Plate stock	Longitudinal	Parallel
B	Plate stock	Longitudinal	Perpendicular
C	Plate stock	Transverse	Parallel
D	Plate stock	Transverse	Perpendicular
E	Affected zone	Longitudinal	Parallel
F	Affected zone	Longitudinal	Perpendicular
G	Affected zone	Transverse	Parallel
H	Affected zone	Transverse	Perpendicular
I	Fusion line	Longitudinal	Parallel
J	Fusion line	Longitudinal	Perpendicular
K	Fusion line	Transverse	Parallel
L	Fusion line	Transverse	Perpendicular
M	Weld	Longitudinal[a]	Parallel
N	Weld	Longitudinal[a]	Perpendicular
O	Weld	Transverse[b]	Parallel
P	Weld	Transverse[b]	Perpendicular

a. Transverse to the welding groove.
b. Longitudinal to the welding groove.

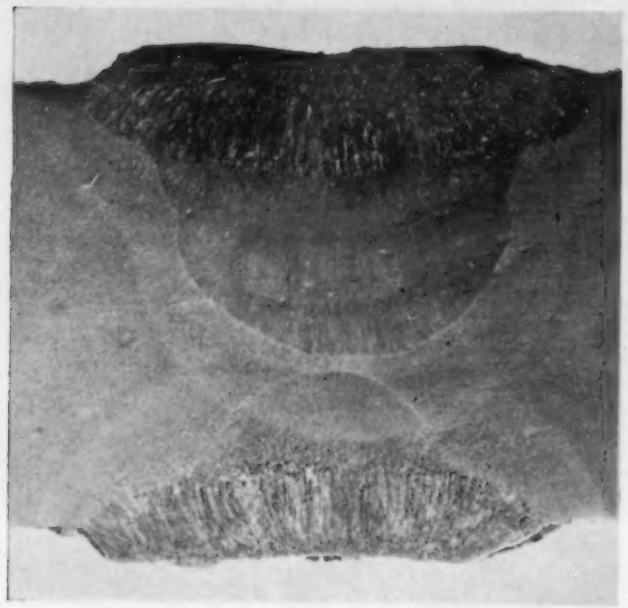
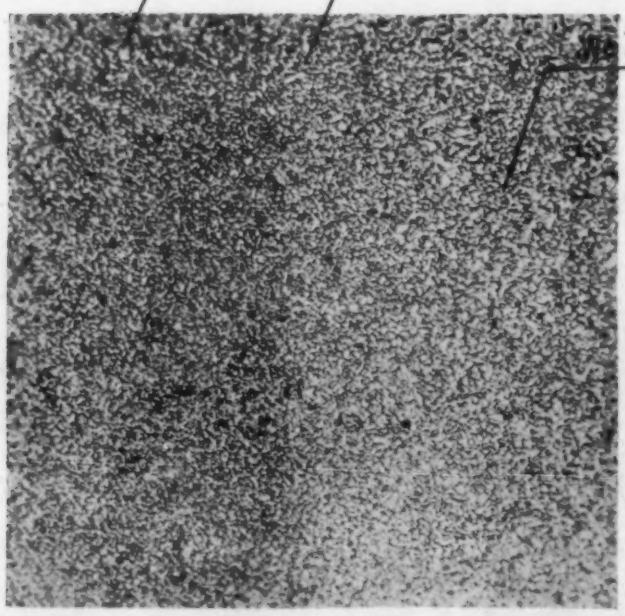


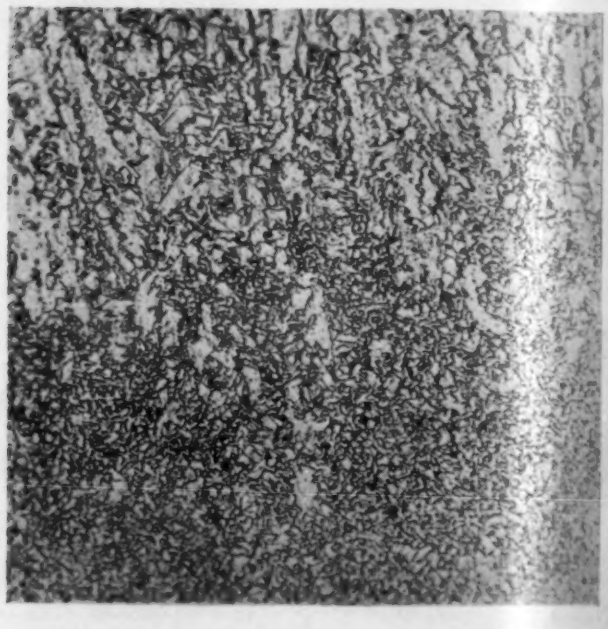
Fig. 13. View A—(Left) Nickel Steel Plate Weld. Etched with 4 per cent nital. Magnification 2 diameters.

View B—(Right) Nickel Steel Plate Stock. Etched with 4 per cent nital. Magnification 100 diameters.

View D—(Below) Weld Metal Nickel Steel Plate Weld. Etched with 4 per cent nital. Magnification 100 diameters.



View C—(Left) Fusion Zone Nickel Steel Plate Weld. Etched with 4 per cent nital. Magnification 100 diameters.



Tests to determine the effect of temperature on the rate of decrease in the impact value of nickel steel were made at 70, 0, —25, —50, and —75 deg. F., and the results of these tests are portrayed graphically in Fig. 14. It will be noted that the impact value falls uniformly over the entire temperature range of from 70 deg. F. to —75 deg. F. Several investigators have reported an anomalous impact behavior of certain steels (including nickel steel) at —25 deg. F., stating that the impact value was as low at this temperature as at —50 deg. F. Tests were made (12 at each temperature) to determine the accuracy of

Table 12.—Summarization of Chemical, Physical, and Impact Test Data on Crane Cast Nickel Steel

(All of the physical and impact test data were obtained on cast nickel steel which had been normalized at 1725 deg. F., renormalized at 1510 deg. F., and drawn at 1200 deg. F.)

Test	Unit	Temp. of Test, deg. F.	Number Heats Represented	Number Tests Represented	Test Values Obtained		
					Min.	Max.	Aver.
Chemical Analysis:							
Carbon	Per Cent	..	47	47	0.14	0.28	0.20
Manganese	Per Cent	..	47	47	0.55	0.71	0.65
Phosphorus	Per Cent	..	47	47	0.010	0.028	0.019
Sulphur	Per Cent	..	47	47	0.004	0.023	0.013
Silicon	Per Cent	..	47	47	0.20	0.39	0.26
Nickel	Per Cent	..	47	47	2.13	2.64	2.40
Physical Tests:							
Tensile Strength	Lb. per sq. in.	70	47	47	69,200	85,200	75,900
Yield Point	Lb. per sq. in.	70	47	47	45,800	63,800	54,700
Elongation in 2 in.	Per Cent	70	47	47	28.5	36.0	32.5
Reduction of Area	Per Cent	70	47	47	50.0	66.4	59.7
Impact Tests:							
Charpy Impact Value	Ft.-lbs.	70	47	92	27.0	47.5	34.5
Charpy Impact Value	Ft.-lbs.	0	12	24	24.0	36.0	28.8
Charpy Impact Value	Ft.-lbs.	—50	47	94	15.0	33.0	24.9
Charpy Impact Value	Ft.-lbs.	—75	47	93	12.0	30.5	22.4

All values obtained by Crane Laboratories.

these statements, but no evidence of anomaly was noted.

To determine the effect of insufficient cropping on the impact value of the nickel steel plates, impact tests were made on specimens from the center of a plate and from a portion of the plate which normally would be crop discard. Approximately a 50 per cent reduction in impact values occurred in the specimen from the crop discard.

In order to determine the effect of carbon content on the impact strength of nickel steel, impact tests were made at 70 deg. F. and —50 deg. F. on specimens taken from five equivalently heat-treated plates of similar nickel content and covering the carbon range of 0.21 to 0.31 per cent. It was found that, above 0.25 per cent carbon, there was some depreciation in impact value, but that at and below 0.25 per cent carbon there was little, if any, change in impact strength.

Nickel Steel Castings

The heat treatment developed for the nickel steel castings was somewhat different from that developed for the rolled plates, due to the persistence of the original "as cast" structure which proved deleterious to the impact value, particularly at the lower temperatures. Table 11 details some of the tests made to determine the optimum heat treatment. It was found that it was necessary to normalize at above 1700 deg. F. to thoroughly eliminate the "as cast" structure, and it was also found that renormalization at approximately 1500 deg. F. refined the grain and im-

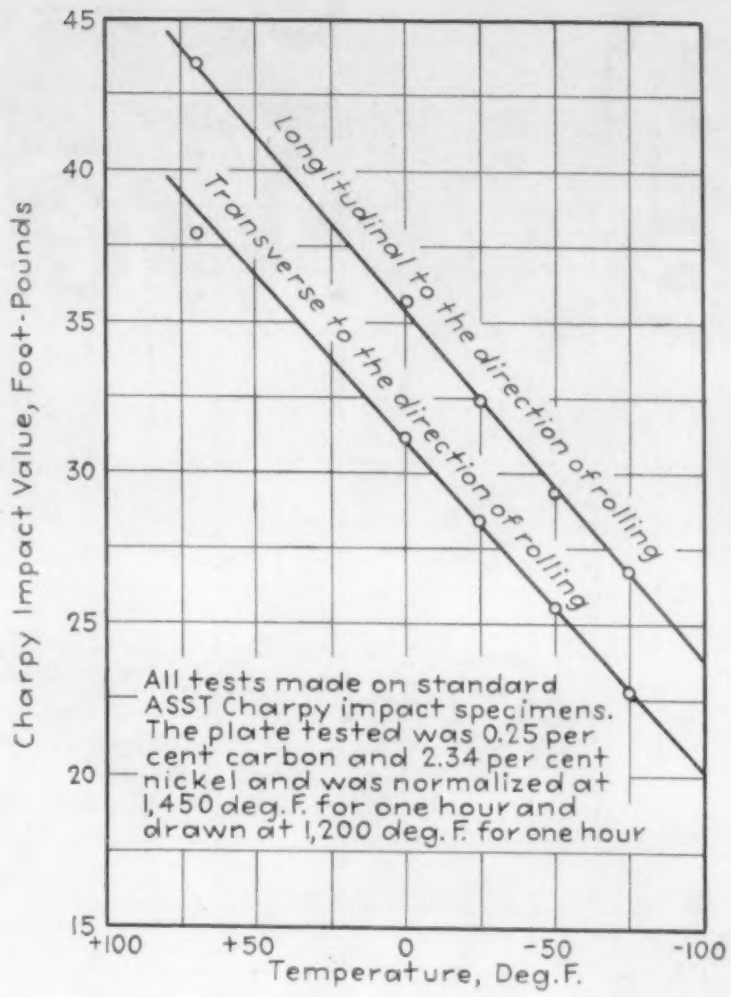
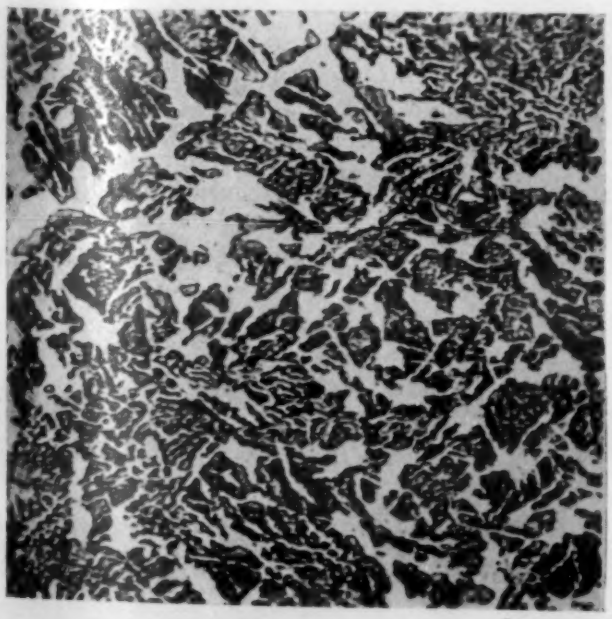

Charpy Impact Value, Foot-Pounds
45
40
35
30
25
20
15
+100
+50
0
-50
-100
Temperature, Deg.F.
Longitudinal to the direction of rolling
Transverse to the direction of rolling
All tests made on standard ASST Charpy impact specimens. The plate tested was 0.25 per cent carbon and 2.34 per cent nickel and was normalized at 1,450 deg. F. for one hour and drawn at 1,200 deg. F. for one hour

Fig. 14. Effect of Temperature on the Impact Strength of Nickel Steel Plates.



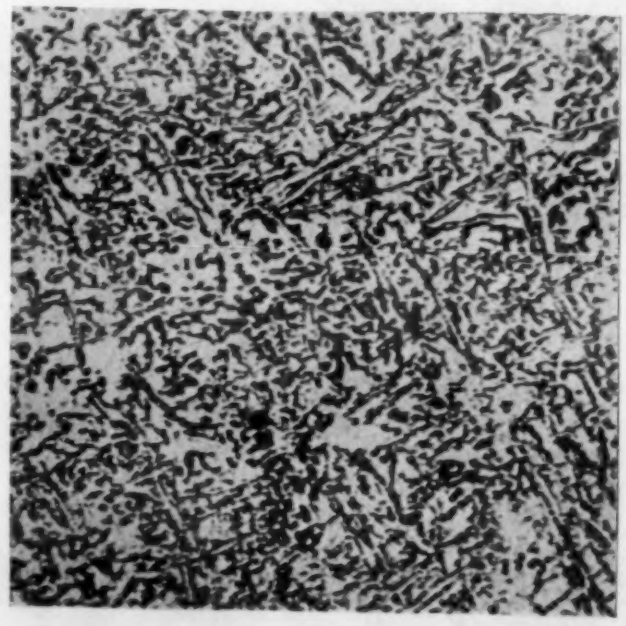
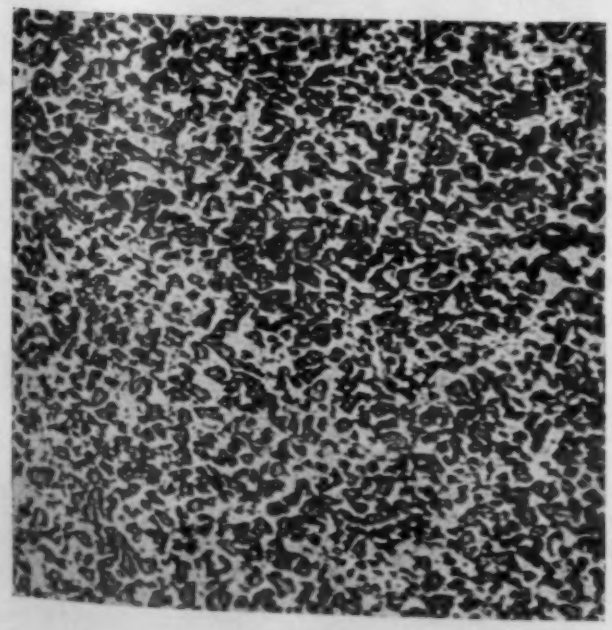
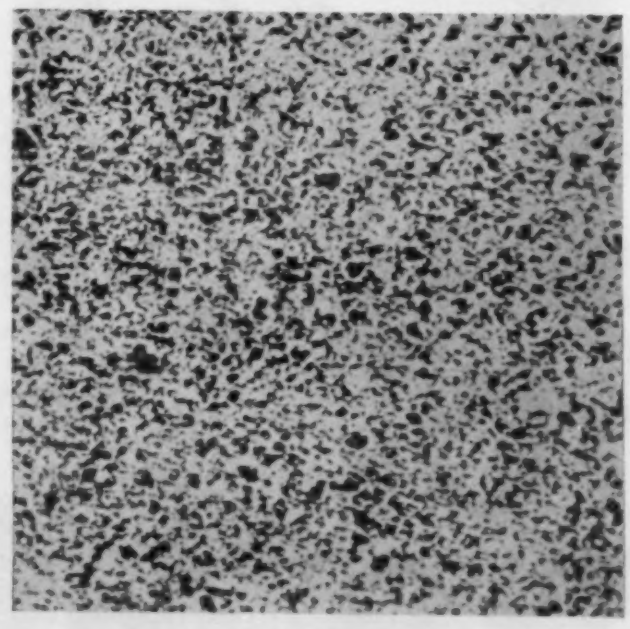


Fig. 15. Effect of Heat Treatment on the Impact Properties and Microstructure of Cast Nickel Steel.

View A—(Left) Cast Nickel Steel As Cast 4.2 ft. lb. at -75 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

View B—(Right) Cast Nickel Steel Normalized at 1450 Deg. F. Drawn 1200 Deg. F. 10.8 ft. lb. at -75 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.





View C (Left)—Cast Nickel Steel Normalized at 1725 Deg. F. Drawn 1200 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

View D—(Right) Cast Nickel Steel Normalized at 1725 Deg. F. Renormalized at 1510 Deg. F. Drawn 1200 Deg. F. 23.0 ft. lb. at -75 Deg. F. Etched with 4 per cent nital. Magnification 100 diameters.

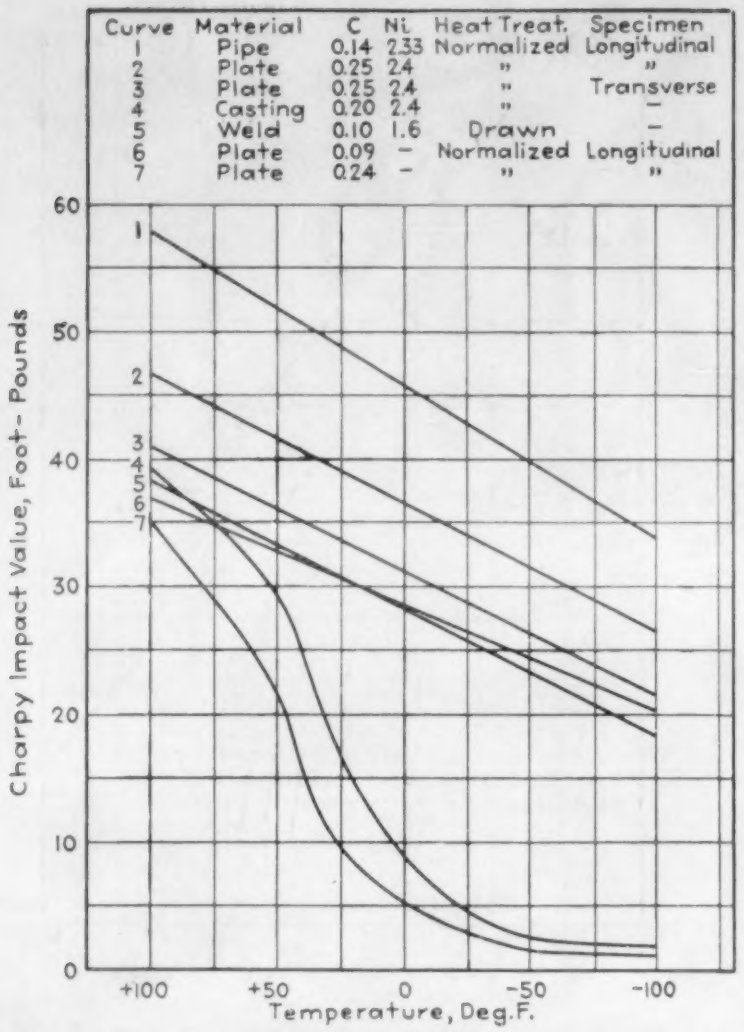


Fig. 16. Effect of Temperature on the Impact Strength of Nickel and Carbon Steel.

proved the impact values. The castings were drawn to 1200 deg. F. after heat treatment to eliminate any residual stresses due to the normalization treatment.

Views A, B, C, and D of Fig. 15 shows the effect of heat treatment on the microstructure of cast nickel steel. Table 12 presents a summarization of the analytical, physical and impact values obtained on 47 heats of castings.

30 mın., and drawing for 2 hrs. at 1200 deg. F. It was found that 1450 deg. F. was slightly too low for effective normalization. This is undoubtedly due to the relatively low carbon content (0.14 per cent) of the steel, the upper thermal critical point of 2.25 per cent nickel steel apparently ascending with decreasing carbon content.

Table 13 shows the results of impact tests made on standard 6-in. nickel steel pipe, on a 2-pass weld, on a 3-pass weld, and on the affected zone of nickel steel pipe. In making these welds, we had two welders each make a weld, and then stress relieved a half (semicircular) of each weld and left the other half in the unrelieved condition. The specimens were taken with their major axis longitudinal to the pipe axis, due to the thinness and curvature of the pipe, and every care was taken to see that the notch was in the weld metal. The tests were all made in triplicate and at —75 deg. F. The two types of modified specimens shown in details D and F of Fig. 1 were used due to the thin wall sections. This also gave a cross check on the correlation curves for these two types of specimens. The values obtained were corrected to the standard 10.0 mm. by 10.0 mm. ASST Charpy impact value from the curves in Fig. 6.

It will be noted that the pipe has nearly twice the impact strength of the weld metal at —75 deg. F., that stress relieving improves the impact strength of the weld metal, but slightly lowers the impact strength of the pipe (which had previously been stress relieved), that the 3-pass weld has better impact strength than the 2-pass weld (probably due primarily to the heat treating effect of subsequently deposited beads), that the pipe stock immediately adjacent to the weld is practically unaffected by the welding, and that the corrected average impact values with the two types of specimens give quite comparable results. Even in the unrelieved condition, reasonably good impact values were obtained on the weld metal.

Fig. 16 shows the effect of temperature on the impact strength of nickel steel plates, nickel steel weld

Table 13.—Effect of Stress Relieving on the Charpy Impact Value at —75 deg. F. of Nickel Steel Pipe and Welds Thereof with Impact Specimens of Different Size and Shape. All Tests Made in Triplicate

Material	Draw Temp., deg. F.	Dimensions of Specimens, mm.			Dimensions Under Notch, mm.			Corrected Charpy Impact Value, Ft.-lbs.		
		Length	Breadth	Thickness	Depth	Breadth	BD[a]	Min.	Max.	Aver.
Pipe	1200	50.8	10	5	5	5	625	33.3	36.8	34.9
	1200	50.8	11.3	5	6.3	5	1250	32.1	37.1	34.6
	....	50.8	10	5	5	5	625	34.7	38.3	36.6
	....	50.8	11.3	5	6.3	5	1250	33.0	40.1	37.3
Pipe Affected Zone	1200	50.8	10	5	5	5	625	30.6	36.1	33.9
	1200	50.8	11.3	5	6.3	5	1250	33.1	38.3	35.0
	....	50.8	10	5	5	5	625	33.9	38.6	36.7
	....	50.8	11.3	5	6.3	5	1250	30.4	38.6	35.1
2-Pass Weld	1200	50.8	10	5	5	5	625	16.9	21.6	19.1
	1200	50.8	11.3	5	6.3	5	1250	15.3	21.7	18.9
	....	50.8	10	5	5	5	625	13.3	19.6	15.9
	....	50.8	11.3	5	6.3	5	1250	14.8	18.7	16.2
3-Pass Weld	1200	50.8	10	5	5	5	625	18.6	23.3	21.2
	1200	50.8	11.3	5	6.3	5	1250	17.4	22.7	20.4
	....	50.8	10	5	5	5	625	14.3	19.2	17.1
	....	50.8	11.3	5	6.3	5	1250	14.8	18.7	16.8

Pipe for Low Temperature Service

The pipe for the low temperature service was all made from one heat of steel which had the following analysis:

	Per Cent
Carbon	0.14
Manganese	0.45
Nickel	2.33

The most satisfactory impact values on this steel were obtained by normalization at 1500 deg. F. for

metal, nickel steel castings, nickel steel pipe, and normalized carbon steel plate. The curves clearly indicate the superiority of 2.25 per cent nickel steel over carbon steel for low temperature impact service.

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